PART 1—Feed Mixing Series

Criteria for Evaluating Feed Mixer Performance

By Dr. Harry B. Pfost
Kansas State University

EDITOR'S NOTE: This article introduces a series of five which, together, will provide the industry with a comprehensive guide which will help any feed manufacturer or mixer determine the quality and efficiency of his mixing operations. All of the articles in the series were researched and prepared by present or former staff members of the department of flour and feed milling industries at Kansas State University, Manhattan.

This first article in the series, by Professor Pfost, constitutes contribution No. 476 of the Kansas Agricultural Experiment Station and is reprinted from the May 9, 1964, issue of Feedstuffs to introduce the series and to provide background information for the following articles.

The second article will discuss a new method of describing particle size. Other articles in the series: "Physical Charactertistics of Feed Microingredients," "Testing Feed Mixtures, Mixers and Related Equipment" and "Testing and Performance of a Vertical Twin Screw Mixer."

Although the importance of good mixing is recognized among animal nutritionists and feed manufacturers, few investigators have reported research results that could be used to correlate degree of mixing with animal growth. A few feeding trials (1) have utilized vitamins A or D, which is unfortunate because potential storage or excess of it in the body would be least likely to cause adverse effects from poor mixing. Until more feeding trials are conducted to show the correlation between degree of mixing and animal health, it will be necessary to set rather arbitrary limits to mixer performance.

Bloom and Livesey ((2) selected as a criterion that 95% of the daily rations received by an animal should contain 90% or more of the daily requirements of the ingredient considered. Merck's Service Bulletin (3) states that a coefficient of variation of less than 5% probably represents a good mix. Bruggemann and Niesar (4) have defined "absolute homogeneity" as a variation no greater than that of the variation of the chemical assay procedure used. None of the authors listed has stated reasons for arbitrarily selecting the values they used.

In solids mixing equipment (5) standard testing procedures for testing mixers are given. Many of the techniques for testing, sampling and evaluating results are applicable to feed mixing equipment.

Several criteria might be considered in arriving at a decision regarding the degree of mixing desired; among them are:

1. The mix should provide each animal with a given percentage of his daily nutrient requirements.
2. It should be adequate to prevent frequent occurrence of toxic levels.
3. It should be adequate to insure that samples will be within limits set by control organizations.
4. Inaccurate sampling or assay techniques.
5. Loss of a material from the mixture, as through dust collector systems.

Since an animal can select only from feed that has been mixed, conveyed and stored, feed manufacturers should be testing their products as they leave the plant. Evaluation of the mixer discharge can determine only the actual mixer performance.

For purposes of discussion here, we may assume that samples that represent about the average daily requirements of an animal consuming the feed are being taken as the feed leaves either mixer or the feed plant.

Since several authors have selected the statistical measure of coefficient of variation, it should be defined

$$V = \frac{s}{m} \times 100$$

V = coefficient of variation in percent.
s = standard deviation of the assay value
m = mean of the assay values

The value of the coefficient of variation is frequently calculated from the formulas

$$s^2 = \frac{X_1^2 + X_2^2 + \cdots + X_N^2 - Nm^2}{N-1},$$

and

$$m = \frac{X_1 + X_2 + \cdots X_N}{N},$$

where:
N = number of samples assayed
Xi = assay value of the i'th sample
m = mean value of all samples

Naturally N must be large enough to minimize errors from too few samples. (In research conducted by the author and associates a minimum of 10 samples usually are taken.)

If a criterion similar to that selected by Bloom and Livesey is elected, then a condition similar to that shown in Figure 1 results. If a wide tolerance limit B is selected, then a larger percentage of the samples fall within limits. Figure 2 shows the effect of coefficient of variation on the probability that a sample will exceed a given tolerance limit. From Figure 2 one can see that the Bloom and Livesey criterion corresponds to a coefficient of variation of approximately 5%.

If it is important that some minimum level of a component should be found in samples taken, then, a different analysis can be made. This problem might occur if a feed carried some minimum guaranteed level and an excess of the component would not be harmful. Figure 3 shows the amount of excess of the component over the guarantee which must be added to insure, with a given probability, that the guaranteed level will be found under conditions of varying coefficients of variation for mixing. From this chart, it is readily evident that better mixing will allow less excess to be used with a low probability of failing to meet the guarantee.

In some cases, a large excess of a component in the daily ration of an animal might be toxic. Figure 4 shows the coefficient of variation that might

FIGURE 1. Tolerance Limits Shown in Relation to a Normal Distribution Curve

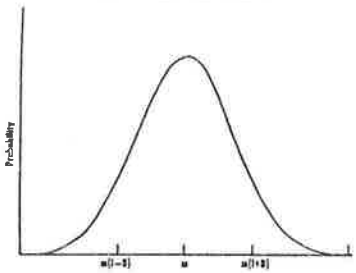


FIGURE 3. Probability of Meeting the Minimum Guarantee of a Component When the Degree of Mixing and Excess Varies

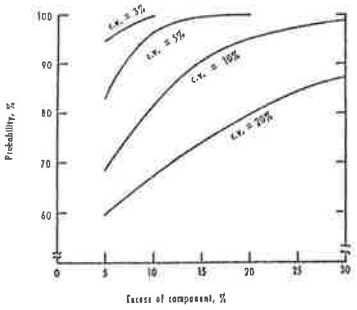


FIGURE 5. Poisson Distribution for an Average of Five Particles Per Sample

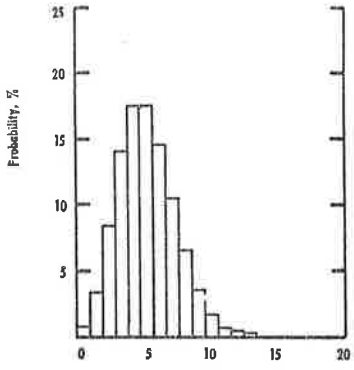


FIGURE 2. Probability of Exceeding a Given Tolerance Under Various Coefficients of Variation in the Mixture

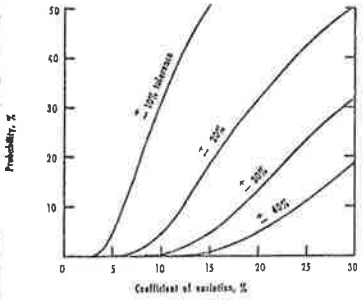


FIGURE 4. Probability of Finding a Large Excess of a Component in a Sample When Mixing is Imperfect

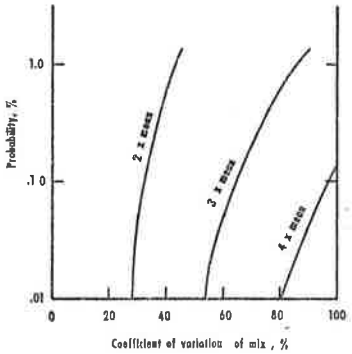
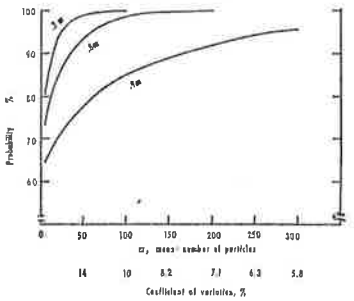


FIGURE 6. Probability of Equalling or Exceeding a Fraction of the Mean as Related to the Number of Particles in a Sample



be allowable for various probabilities and excesses. For example, if it is desired that the probability will not exceed 0.001 (0.1%) that a level of "2x mean" will be found in a sample, then the coefficient of variation should be less than about 32%.

Variations of the level of a component in a sample can occur if the number of particles of the component expected in the sample is not very large. When a limited number of particles are distributed among various sample spaces, the probability of finding a given number of particles, x, in a particular sample space is given by the Poisson distribution formula,

$$P(x) = \frac{m^x}{x!e^m}$$

where:

P(x) = probability of x particles in a sample

m = mean number of particles per sample

e = base of natural logarithms

Figure 5 shows the distribution of the number of particles that may be expected when m equals 5.

The Poisson distribution has certain fixed properties that lead to the relationship

$$V = \frac{100}{\sqrt{m}}$$

For example, if there were an average of 100 particles per sample, then the coefficient of variation would be 10%. Hence, even if the mixer operated "perfectly," there would still be a coefficient of variation of 10% among samples.

Figure 6 shows the effect of number of particles per sample on the probability that any particular sample will contain a given fraction, or more, of the average amount of the component.

Since some drugs have been reported to be toxic at levels of twice normal feeding levels (6), it probably would not be safe to exceed a coefficient of variation of about 20% (Figure 4). The coefficient of variation of 5% selected by some authors appears to be conservative.

New Standard Deviation

Many feed manufacturers say they have no mixing problems because their feed meets the requirements of state feed control inspection. It should be noted that most state feed control assays are made on samples composited from 10 or more individual samples. When samples are composited, the new standard deviation is given by

$$\bar{s} = \frac{s}{\sqrt{n}}$$

Where s = standard deviation of individual samples

n = number of individual samples composited

Hence the coefficient of variation of a composited sample would become

$$\bar{V} = \frac{V}{\sqrt{n}}$$

For example, a feed might have a true coefficient of variation of 30% where double levels of a component would occur more frequently than 0.01% of the time. If 10 samples were composited, the coefficient of variation would appear to be less than 10%, $30/\sqrt{10}$, which would seem to be quite safe and would exceed a 10% tolerance limit less than 30% of the time as shown in Figure 2. The general effect and purpose of composited samples has been to secure an accurate measure of the average level of components—not to measure variations resulting from processing and handling.

Summary

From the results of this theoretical analysis it appears that the total coefficient of variation should not exceed 20%, to avoid possible toxic effects. If usual numbers of particles of a component are present per sample, then mixer tolerance cannot be so broad. Probably with most components the coefficient of variation that may be allowed due to poor mixing and/or segregation could range as high as 5-10%.

REFERENCES CITED

(1) Creger, C. R.—A Study of Distribution of Microingredients in Mixed Feeds; unpublished M. S. Thesis, Kansas State University, 1957.

(2) Bloom, C., and Livesey, E. F.—Particle Sizes for Additives to Animal Foods; Mfg. Chemist, 24:37-375, September, 1953.

(3) Anon.—Microingredients in Feeds; Merck & Co., Inc., Highway, N.J., 1959.

(4) Bruggemann, J., und Nieser, H.—Der Einfluss der morphologischen Beschaffenheit der Mischkomponenten auf den Homogenitatsgrad einer Mischung, Fachverbandes der Futtermittel Industrie, Hamburg, West Germany, 1962.

(5) Anon.—Solids Mixing Equipment: Standard Testing Procedure, Am. Inst. of Chemical Engineers.

(6) Anon.—FDA Cites Actions Against Feed Firm; Feedstuffs, Vol. 33:1, 7, Sept. 2, 1961.

Reprinted from FEEDSTUFFS *of October 29, 1966*
(Vol. 38, No. 43, p. 30)

PART 2—Feed Mixing Series

Describing Particle Size Distribution Of Feedstuffs Statistically

By Dr. Verl Headley and Dr. Harry Pfost
Kansas State University

EDITOR'S NOTE: This article is the second in a series of five which, together, will provide the industry with a comprehensive guide that will help any feed manufacturer or mixer determine the quality and efficiency of his mixing operation. All of the articles in the series were researched and prepared by present or former staff members of the department of flour and feed milling industries at Kansas State University, Manhattan.

This second article in the series, by Dr. Verl Headley and Dr. Harry Pfost, constitutes contribution No. 555 of the Kansas Agricultural Experiment Station. Other articles in the series will be: "Physical Characteristics of Feed Microingredients," "Testing Feed Mixtures, Mixers and Related Equipment," and "Testing and Performance of a Vertical Twin Screw Mixer."

The present standard methods of describing the particle size distribution of ground feed materials lack simplicity and versatility in many respects. Analyses of sieving data by these methods are lengthy and difficult to compare with different samples in relation to other important aspects, such as surface area or number of particles. This method which has been used by the American Society of Agricultural Engineers and American Society of Animal Production employs a number weighting procedure of sieving data; the procedure yields two moduli, a modulus of fineness and modulus of uniformity (1). These two moduli may be used to describe the fineness of grind and denote the range of particle size. More informative and effective methods for describing particle size distributions have been used in other industries for various materials. This article illustrates the theory and applications of small particle statistics to feed materials.

Samples from a population may sometimes represent a normal distribution described mathematically by the relationship:

$$f(x) = \frac{1}{\sigma(2\pi)^{0.5}} \exp -1/2 \left(\frac{X-\mu}{\sigma}\right)^2 \quad (1)$$

Normally, $\bar{X} = \frac{\Sigma x_i}{n}$ and (2)

$$s^2 = \frac{\Sigma(X-\bar{X})^2}{n-1} = \frac{\Sigma x^2 - \frac{(\Sigma X)^2}{n}}{n-1} \quad (3)$$

are computed as estimates of μ and σ^2 respectively, with the square root of the variance yielding the standard deviation (2). A distribution on which these parameters are illustrated is shown in Figure 1. For a normal distribution, the probability of randomly selecting an observation within intervals about the mean is given by:

$$P(\mu - \sigma \leq X \leq \mu + \sigma) = 0.68 \quad (4)$$

$$P(\mu - 2\sigma \leq X \leq \mu + 2\sigma) = 0.95 \quad (5)$$

When incremental values under the probability curve are summed,

$$F(X) = \int_{-\infty}^{x} f(x)\,dx, \quad (6)$$

a cumulative distribution curve similar to the one shown in Figure 2, is obtained.

Distribution Data

Arithmetic probability paper facilitates the plotting of cumulative distribution data yielding the graphical solutions for $\bar{X}$ and S. When a true normal distribution exists, then an arithmetic probability plot will yield a linear relationship as shown in Figure 3. The value of the mean, $\bar{X}$, is determined as the value of X at a probability of 0.5 and the standard deviation, S, by differences where

$$S = (\bar{X} - X_{16}) = (X_{84} - \bar{X}). \quad (7)$$

The particle size distributions of some ground materials, usually by weight (or volume) or by count, are found to be skewed or non-normal as indicated by Figure 4. With some distributions, the logarithm of the independent variable, particle size, may be plotted along the abscissa, resulting in a curve shape similar to that of a normal distribution as shown in Figure 5. When this situation exists, it is said to be a log-normal distribution. By the same token, the plotting of cumulative distribution data versus the logarithm of particle size or diameter on log probability paper will yield a linear relationship as shown in Figure 6.

The log-normal distribution function may be expressed as:

$$f(d)d(d) = \frac{1}{\ln \sigma_{gn}(2\pi)^{0.5}} \exp -1/2 \left(\frac{\ln d - \ln \mu_{gn}}{\ln \sigma_{gn}}\right)^2 d(\ln d). \quad (8)$$

The value of the geometric mean particle size or diameter of the distribution by number of particles, d_{gn}, of a sample may be estimated by the relationship—

$$\ln d_{gn} = \frac{1}{n}(\ln d_1 + \ln d_1 + \cdots + \ln d_n) \quad (9)$$

with the symbol, n, signifying the number of particles (3,4,5). Similarly, the geometric standard deviation by number, S_{gn}, of a sample may be estimated by:

$$\ln S_{gn} = \left[\frac{\sum_{i=1}^{n} (\ln d_i - \ln d_{gn})^2}{n}\right]^{0.5}, \quad (10)$$

This technique could be applied when each particle is measured individually as in a microscopic examination.

FIGURE 1. Curve for a Normal Frequency Distribution

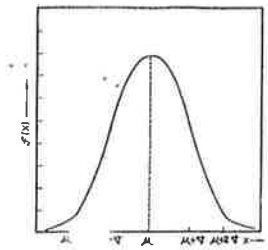


FIGURE 3. Arithmetic Probability Curve for a Cumulative Normal Distribution

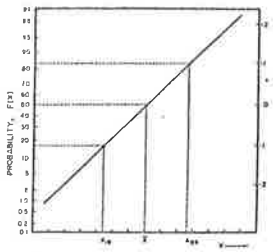


FIGURE 2. Curve for a Cumulative Normal Distribution

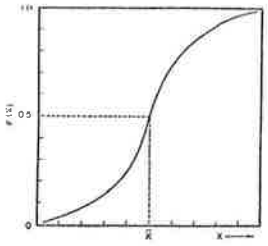
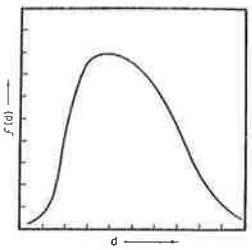


FIGURE 4. Curve for a Non-Normal Frequency Distribution



Frequently, when making particle size determinations, it is more convenient to weigh or measure the volume of a sample within a given size range, as is the case with sieving or sedimentation assays (6). In this case, the log-normal particle size distribution may be expressed as:

$$f(d)\,d(d) = \frac{1}{\ln \sigma_{gw}\,(2\pi)^{0.5}} \exp -1/2 \left(\frac{\ln d - \ln \mu_{gw}}{\ln \sigma_{gw}}\right)^2 d(\ln d), \quad (11)$$

The parameters for a sample may now be estimated by:

$$\ln d_{gw} = \frac{\sum (W_i \ln d_i)}{\sum W_i}, \text{ and} \quad (12)$$

$$\ln S_{gw} = \left[\frac{\sum (W_i (\ln d_i - \ln d_{gw})^2)}{\sum W_i}\right]^{0.5} \quad (13)$$

In equations (9) through (13) it is arbitrary as to what base the logarithms are taken. Natural logarithms may be used or base 10 may be more convenient.

Log Probability

Log probability paper may also be used to obtain solutions graphically for the above parameters. When cumulative sieving data are plotted by weight along the probability scale (ordinate) versus the logarithm of the particle size along the abscissa, the values for the distribution parameters may be read directly. The geometric mean diameter of the distribution by weight may be read as the value d_{gw}, at the 50% probability point, and the geometric log-normal standard deviation, S_{gw}, as

$$S_{gw} = \frac{d_{84}}{d_{gw}} = \frac{d_{gw}}{d_{16}} \quad (14)$$

where d_{84} and d_{16} are the particle diameters corresponding to the probabilities of 16% and 84% respectively.

Similarly, if the estimated parameters, S_{gw} and d_{gw}, are known, the log probability distribution plot may be reconstructed and the probability of finding a particular fraction in the given size range, d_1 to d_2, may be read directly as $F_w(d_2) - F_w(d_1)$.

In many cases it is desirable to know the total exposed surface area and total number of particles in a particular distribution. The following equations have been derived by the authors using log-normal distribution parameters by weight, which yield solutions predicting both total surface area and number of particles.

If the total sample weight of a material is known, the weight in the i'th interval, by sieving for example, is:

$$W_i = W_t (F_w(d_u) - F_w(d_o)) \quad (15)$$

and the number of particles in the i'th interval will be:

$$N_i = \frac{W_i}{\rho \beta_v d_i^3} \quad (16)$$

Similarly, the interval surface area may be calculated as:

$$A_{si} = N_i \beta_a d_i^2 \quad (17)$$

Replacing the value of N_i in the interval surface area equality, the relationship takes the form:

$$A_{si} = \frac{\beta_a W_i}{\beta_v \rho d_i} = \frac{\beta_a W_t (F_w(d_u) - F_w(d_o))}{\beta_v \rho d_i} \quad (18)$$

The weight probability within an interval may be calculated by the relationship:

$$F_w(d_2) - F_w(d_1) = \frac{1}{\ln \sigma_{gw}\,(2\pi)^{0.5}} \int_{d=d_1}^{d=d_2} \exp -1/2 \left(\frac{\ln d - \ln \mu_{gw}}{\ln \sigma_{gw}}\right)^2 d(\ln d) \quad (19)$$

Thus, the integral form for predicting the total surface area for a particular log-normal distribution is:

$$A_{st} = \frac{\beta_a W_t}{\beta_v \rho \ln \sigma_{gw}\,(2\pi)^{0.5}} \int_{d=0}^{d=\infty} \frac{1}{d} \exp -1/2 \left(\frac{\ln d - \ln \mu_{gw}}{\ln \sigma_{gw}}\right)^2 d(\ln d) \quad (20)$$

Introducing $Z = \ln d$ (the logarithm base now must be the natural to base e) and integrating, the equation for total surface area becomes:

$$A_{st} = \frac{\beta_a W_t}{\beta_v \rho} \exp (0.5 \ln^2 \sigma_{gw} - \ln \mu_{gw}) \quad (21)$$

Manipulation of the exponential also yields an equivalent expression for total surface area as:

$$A_{st} = \frac{\beta_a W_t \sigma_{gw}^{(\ln(\sigma_{gw})^{0.5})}}{\beta_v \rho \mu_{gw}} \quad (22)$$

Similarly, when a given distribution by weight is known, it may be desirable to know the total number of particles. The equation for predicting the total number of particles in a particular log-normal distribution may be derived from the basic interval equation,

$$N_i = \frac{W_t (F_w(d_u) - F_w(d_o))}{\rho \beta_v d_i^3} \quad (23)$$

The integral form by introducing the probability distribution becomes:

$$N_t = \frac{W_t}{\rho \beta_v \ln \sigma_{gw}\,(2\pi)^{0.5}} \int_{d=0}^{d=\infty} \frac{1}{d^3} \exp -1/2 \left(\frac{\ln d - \ln \mu_{gw}}{\ln \sigma_{gw}}\right)^2 d(\ln d) \quad (24)$$

Integration and simplification yield an equation for the total number of particles as:

$$N_t = \frac{W_t}{\rho \beta_v} \exp (4.5 \ln^2 \sigma_{gw} - 3 \ln \mu_{gw}) \quad (25)$$

By the same token manipulation of the exponential yields an equivalent expression for total number of particles as:

$$N_t = \frac{W_t \sigma_{gw}^{(\ln(\sigma_{gw})^{4.5})}}{\beta_v \rho \mu_{gw}^3} \quad (26)$$

Therefore, from known values for both surface and volume shape factors, the specific weight of a material, its sample weight, log-normal geometric

FIGURE 5. Semilogarithmic Curve of a Non-Normal Frequency Distribution

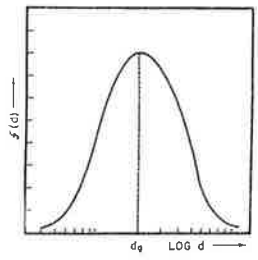


FIGURE 7. Theoretical Curves Indicating the Application of Transformation Equations

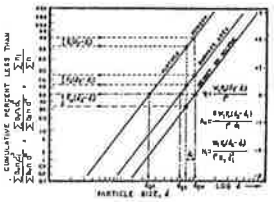


FIGURE 8. Comparison for Normal or Log-Normal Distribution for Milo Ground Through 1/8 In. Screen

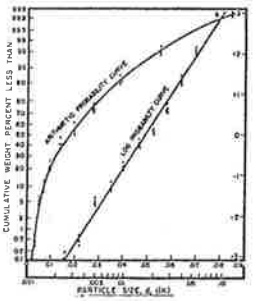


FIGURE 6. Log Probability Curve of a Log-Normal Distribution

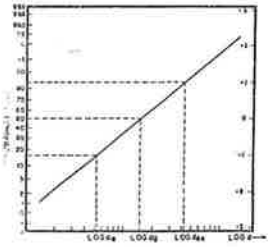


FIGURE 9. Log Probability Distribution Curves for Straight Grade Flour, Vitamin A and Soybean Meal

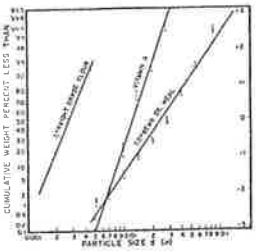


FIGURE 10. Log Probability Distribution Curves for Corn and Milo Ground Through ⅛ In. Screen

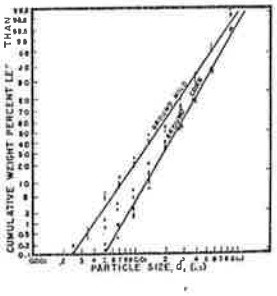


FIGURE 11. Log Probability Distribution Curve for Whole Oats Ground Through a ⅛ In. Screen

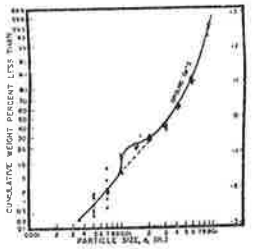


FIGURE 12. Typical Data Sheet Used for Tabulation of Sieving Data and Calculation of Log-normal Particle Size Distribution Parameters by Weight

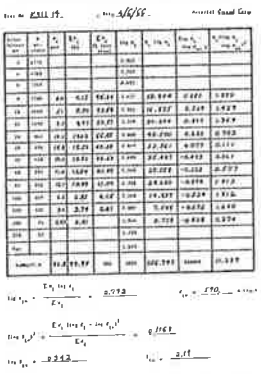


FIGURE 13. Cumulative Particle Size Distribution, by Weight, for a Ground Corn Sample

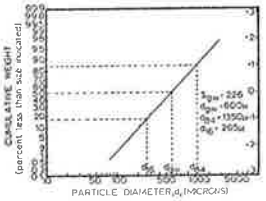


FIGURE 14. Cumulative Particle Size Distribution by Weight, Surface Area and Number for a Ground Corn Sample

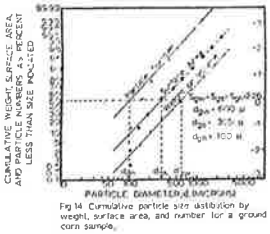


Fig 14 Cumulative particle size distribution by weight, surface area, and number for a ground corn sample.

standard deviation by weight, and geometric mean particle diameter by weight, one may apply the above derived equations predicting the total exposed surface area or number of particles in a particular sample which is log-normally distributed.

If a sample of ground material is log-normally distributed, a complete graphical representation of the respective particle size distributions by weight (or volume), surface area and particle numbers may be drawn on log probability paper when weight distribution parameters σ_{gw} and μ_{gw}, are known, by applying suitable transformation equations (5). The transformation equations yield the necessary respective parameters of geometric mean particle size (or diameter), log-normal geometric standard deviation for both surface area and particle number distributions from weight distribution parameters. To obtain these equations requires first a series of integrations of the exponential log-normal distribution equation for the first, second, third and fourth moments of particle size weighted by particle number, the first moment of particle size weighted by surface area, and the first moment of particle size weighted by volume (or weight). Then by substitution of integration equalities, making use of the unique characteristic that the log-normal standard deviations by weight (or volume), surface area and number are equal,

$$\sigma_{gw} = \sigma_{gs} = \sigma_{gn} \qquad (27)$$

the following transformations are obtained:

$$\ln \mu_{gs} = \ln \mu_{gw} - \ln^2 \sigma_{gw} \qquad (28)$$

and

$$\ln \mu_{gn} = \ln \mu_{gw} - 3 \ln^2 \sigma_{gw} \qquad (29)$$

Log-Normal Distribution

Once these parameters are obtained, the theoretical log-normal distribution curves by surface area and particle number may be immediately constructed. These distribution curves drawn on log probability paper will be both linear and parallel to the existing log-normal weight distribution curve as shown in Figure 7.

The logical question now is: are ground feeds and feed ingredients normally distributed or log-normally distributed? Experimental and statistical implications indicate neither, since rarely, if ever, is any distribution truly normally distributed, and by the same token rarely, if ever, is any distribution truly log-normally distributed. The decision may be made by asking which type of distribution ground feed materials follow more closely. A cumulative arithmetic probability plot of a truly normal distribution yields a straight line, and, similarly, a cumulative log probability plot of a truly log-normal distribution also yields a linear relationship. Thus, Figure 8 indicates that ground sorghum grain approaches more closely that of a log-normal distribution.

In an attempt to confirm this decision, further sieving tests were performed on a series of other feed materials and their particle size distributions plotted on log probability paper as shown in Figures 9, 10 and 11. Figure 11 indicates that extremely nonhomogeneous materials such as oats are difficult to describe; the grinding characteristics of the hull and groat are quite different.

Application to Sieving Data

In order to use this method satisfactorily, it is desirable to select a sufficient number of sieves of a standard series which will provide the complete weight distribution (minimum of six sieves recommended). Figure 12 illustrates a data sheet which has been used by the authors as a combination data and calculation sheet. This sheet was prepared using sieve openings in microns; thus, the logarithm of the particle size (or diameter) to the base 10 becomes a convenient positive number. The average particle size on a sieve is calculated as the geometric mean diameter of the sieves through which the particles passed and upon which they were retained.

$$d_i = (d_n \cdot d_o)^{1/2} \qquad (30)$$

Figure 12 illustrates typical sieving data for a 100 gram sample of ground corn. The weight of material on each sieve is shown as W_i. The percent retained on each sieve is calculated and should total approximately 100%, depending upon rounding errors. Other calculations are made to determine the geometric mean particle diameter, d_{gw}, and geometric mean standard deviation, S_{gw}.

The summed percentages from Figure 12 have been plotted on logarithmic probability paper as shown in Figure 13. Note that in plotting the data the value of 0.41% which was retained on the 200 sieve was plotted as 0.41% less than a size equivalent to number 150 sieve (105 microns). From Figure 13 the geometric mean particle diameter was determined as 600 microns compared to a calculated value of 590 microns. Similarly, the plotted values were used to determine the log-normal geometric standard deviation of

$$S_{gw} = \frac{600}{265} = 2.26$$

which compares closely to the calculated value of 2.19.

The values obtained can be used to calculate the total surface area of a sample or total number of particles in a sample. At this point it is necessary to convert the geometric mean particle size (or diameter) to centimeters, or $d_{gw} = 0.0590$ cm. if sample weight is expressed in grams and specific weight in grams per cubic centimeter.

If we assume that the particles are cubical then $\beta_s = 1$ and $\beta_s = 6$. (If spheres are assumed then $\beta_v = \frac{1}{6}\pi$ and $\beta_s = \pi$). Also, we may assume that the specific weight is approximately 1.4 gm./cm³ for this material.

The total surface area of one gram of this material can be calculated by equation (21 or 22) as

$$A_{st} = \frac{6 \times 1}{1 \times 1.4} \exp \left(0.5 (\ln 2.19)^2 - \ln .0590\right)$$

$$= 4.28 \exp \; 0.5 (0.784)^2 - (-2.82)$$

$$= 4.28 \exp (3.13)$$

$$= 98. \; cm^2$$

Similarly, the number of particles per one gram sample may be calculated by equations (25 or 26) as

$$N_t = \frac{1}{1 \times 1.4} \exp (4.5 (\ln 2.19)^2 - 3 \ln .0590)$$

$$= .714 \exp \; 4.5(0.784)^2 - 3(-2.82)$$

$$= .714 \exp (11.22)$$

$$= 53{,}400 \text{ particles}$$

Also the log-normal distribution parameters and curves for both surface area and particle number distributions for the ground corn sample may be estimated from the weight distribution parameters d_{gw} and S_{gw}, and through application of transformation equations which determine the geometric mean particle sizes d_{gs} and d_{gn}. A unique characteristic of log-normal distribution is that

$$S_{gw} = S_{gs} = S_{gn},$$

This indicates that the weight (or volume) surface area, and particle number distribution curves for the sample will be parallel, since for all practical purposes the log-normal geometric standard deviation determines the slope of the distribution curves. Inserting the weight distribution parameter values of 600 microns and 2.26 for d_{gw} and S_{gw} as obtained of Figure 13 into equations 28 and 29 the geometric mean particle sizes (or diameters) d_{gs} and d_{gn} are calculated at 308 microns and 82 microns respectively. These distribution parameters compare quite well to those obtained in Figure 14 where the distributions were

determined by long hand for comparison through application of incremental equations 16 and 17. Thus, the transformation equations may be used to calculate d_{es} and d_{ea} from the original weight distribution parameters d_{ew} and S_{ew} obtained through sieving. These parameters will in turn permit the graphical representation of weight (or volume), surface area, and particle number distribution curves to be obtained quickly and effectively. By the same token one may use the log-normal probability-particle size distribution curves to estimate both total number of particles and total surface area in a sample of known weight.

Summary

A method for describing the particle size distribution of feed materials by small particle statistics is presented.

Equations are derived on the basis that the distributions of many ground feed materials are log-normal. Methods for determining the log-normal particle size weight distribution parameters of geometric mean particle size and geometric standard deviation are demonstrated. Equations are derived containing these parameters for calculating total surface area and total number of particles in a particular sample.

Transformations of the log-normal particle size distribution parameters by weight to those for surface area and number of particles are illustrated mathematically and the corresponding distributions graphically.

The results of actual particle size analysis data obtained by sieving indicate that those ground feed materials which are fairly homogeneous have particle size distributions which can be represented log-normally.

Application of this method of small particle statistics to actual sieving data are shown. Use of a typical data sheet, determination of the necessary log-normal parameters, and total distribution for number of particles, surface area and weight (or volume) are illustrated both mathematically and graphically.

It is believed that these methods presented provide for greater flexibility in the use of particle size data than do fineness modulus and modulus of uniformity.

DEFINITIONS OF SYMBOLS

A_{si} = surface area of particles in the i'th interval
A_{st} = total surface area of particles
β_s = shape factor for calculating surface area of particles
β_v = shape factor for calculating volume of particles
d = particle size or diameter
d_i = particle diameter in the i'th interval
$d_i = (d_o \cdot d_u)^{0.5}$, the particle size or diameter in the i'th sieve interval
d_o = size of sieve opening through which particles will not pass
d_u = size of adjacent sieve opening through which particles will pass
d_{en} = geometric mean particle size or diameter by particle number distribution of sample
d_{es} = geometric mean particle size or diameter by surface area distribution of sample
d_{ew} = geometric mean particle size or diameter by weight distribution of sample
$f(x)$ = probability density or frequency of x
$f(d)$ = probability density or frequency of d
$F(x)$ = probability of cumulative distribution of X
$F_w(d)$ = probability of cumulative weight distribution of d
N_i = number of particles in the i'th interval
N_t = total number of particles in sample
n = number of particles denoting parent population
ρ = specific weight of material
S = standard deviation of sample estimate
S_{en} = geometric log-normal standard deviation of sample estimate by particle number distribution
S_{es} = geometric log-normal standard deviation of sample estimate by surface area distribution
S_{ew} = geometric log-normal standard deviation of sample estimate by weight distribution
σ = standard deviation of the parent population
σ_{gn} = geometric log-normal standard deviation of parent population by number distribution
σ_{gs} = geometric log-normal standard deviation of parent population by surface area distribution
σ_{gw} = geometric log-normal standard deviation of parent population by weight distribution
μ = mean particle size or diameter of parent population
μ_{gn} = geometric mean particle size or diameter of parent population by particle number distribution
μ_{gs} = geometric mean particle size or diameter of parent population by surface area distribution
μ_{gw} = geometric mean particle size or diameter of parent population by weight distribution
X = sample value or size
$\overline{X}$ = arithmetic mean of sample values
π = constant, 3.14
ln = indicates logarithm to base e
log = indicates logarithm to base 10

REFERENCES

1. Anonymous, 1965—Method of determining modulus of uniformity and modulus of fineness of ground feed; Agricultural Engineers Yearbook, p. 378.
2. Dixon, J. W., and Massey, F. J., Jr., 1957—Introduction to Statistical Analysis; McGraw Hill Book Co., 488 pp.
3. Hatch, T., and Choate, S., 1929—Statistical description of the size properties of non-uniform particulate substances; Journal Franklin Institute, March, pp. 369-387.
4. Hatch, T., 1933—Determination of average particle size from the screen analysis of non-uniform particulate substances; Journal Franklin Institute, January, pp. 27-37.
5. Herdan, G., 1960—Small Particle Statistics; Butterworth and Co., Ltd, 418 pp.
6. Whitby, K. T., 1958—The mechanics of fine sieving, Symposium on Particle Size Measurement; American Society of Testing Materials, pp. 3-24.

Reprinted from FEEDSTUFFS *of November 5, 1966*
(Vol. 38, No. 44, p. 50)

PART 3—Feed Mixing Series

Physical Characteristics Of Feed Microingredients

By Dr. Harry Pfost, Dr. Charles Deyoe, Carl Stevens and Edward Morgan

Kansas State University

The physical characteristics of feed ingredients in relation to problems of mixing has received relatively little attention in the literature. Most cereal grains and protein supplement products have approximately the same densities, and, following grinding (not rolling), the ranges of particle sizes are not widely different. Some mineral ingredients have been very finely ground to facilitate assimilation, and the density of most minerals is much higher than that of other feed ingredients. Because the tolerance for day-to-day variation in mineral intake is large for most animals, mixing variations may not be of too great importance.

With the widespread adoption of a wide variety of microingredients after World War II, some authors, such as Bloom and Livesey (1), began to recognize that the number of particles contained in a sample representing the average daily intake of an animal is important. Because of the cost and nutritional importance of these new microingredients, much more attention was given to problems of mixing them. Drug and feed manufacturers have studied the problems of mixing premixes and complete feeds containing critical microingredients; however, a review of the literature discloses little published information regarding the physical properties of feed additives.

EDITOR'S NOTE: This article is the third in a series of five by members of the faculty of the department of flour and feed milling industries, Kansas State University, Manhattan, Kans. Dr. Pfost is a professor, Dr. Deyoe an associate professor, Mr. Stevens a former agricultural extension specialist and Mr. Morgan a former graduate research assistant. The article constitutes contribution No. 554 of the Kansas Agricultural Experiment Station.

Physical properties which might be expected to influence mixing include:

1. Particle size
2. Particle shape
3. Specific weight
4. Hygroscopicity
5. Susceptibility to electrostatic charges
6. Adhesiveness of the particles due to physical properties, such as rough surfaces, or additions of adhesives such as oils.

Many microingredients are not commonly distributed in their pure or most concentrated form. When such microingredients are mixed with another material for distribution the practical effect may be to change the physical properties, relative to mixing, of the original microingredient. The authors have defined two terms to describe the inert, non-active material:

Diluent — an inactive or inert ingredient mixed with an active microingredient for the purpose of diluting the concentration of the active ingredient.

Carrier — an inactive or inert ingredient mixed with an active ingredient for the purpose of changing the practical physical properties of the active ingredient.

When a diluent is used with a microingredient, the mixing properties of the original ingredient will not be drastically altered. The number and size of the original active particles will remain unchanged for all practical purposes. The use of a diluent may facilitate weighing or increase the rate of mixing.

When a carrier is used with microingredient the mixing properties will be drastically altered. For example, if

FIGURE 1. Air Pycnometer

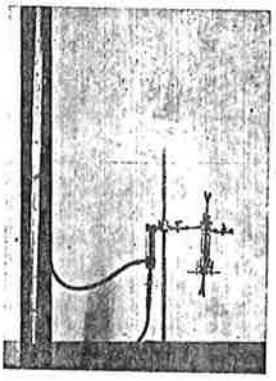
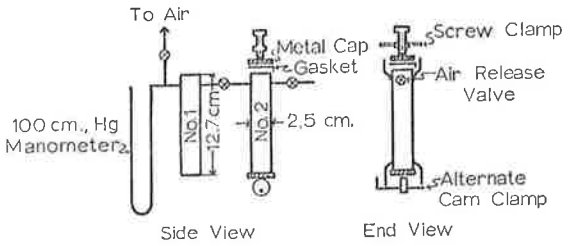


FIGURE 2. Schematic View of Air Pyconometer



a high density drug were finely divided and mixed with an oil and ground grain, the resulting mixture would be distributed in a mixer in about the same way the ground grain would be distributed; or if a liquid microingredient is mixed with a ground grain, the physical properties of the resulting mixture will probably closely resemble the physical properties of the ground grain.

Bruggemann and Niesar (2) have reported the results of particle size measurements of approximately 12 microingredients and 12 common feed ingredients. In an attempt to gain more information regarding the probable mixing properties of a variety of microingredients, the authors obtained samples of a large number of microingredients and measured particle size and density of the materials. In general, the data reported here represents measurements of the microingredients, with or without carrier or diluent, in the most concentrated form in which it is generally distributed to feed or premix manufacturers.

Particle size was measured by sieving techniques if possible. Fineness Modulus and Modulus of Uniformity were used as methods to measure the particle size. These methods are described in the **Agricultural Engineers Yearbook** (3) and **1961 Feed Production Handbook** (4). Geometric mean and geometric standard deviation were also used; these terms are discussed by Headley and Pfost (5).

Density Determinations

Density determinations were made in an air pycnometer shown in Figures 1 and 2. This equipment is used as follows:

The empty volume of each chamber is measured or determined.

A weighed sample of the ingredient is placed in the test cylinder.

The valve between the two cylinders is closed and the valve to the atmosphere on the test cylinder is opened.

Air is admitted to the cylinder No. 1 and the pressure is raised to the maximum P_1. (The authors used a pressure of about 32 cm. of mercury.) The valve to the air source is then closed.

The valve to the atmosphere at the test cylinder is closed and the valve between the cylinders is opened and the final pressure, P_f, is read on the manometer.

The volume of test material is calculated as follows:

Referring to Figure 2 let:

h_1 = Original manometer reading in cylinder 1

$h_2 = h_a$ = Pressure of atmosphere

h_f = Final manometer reading

P_1 = Original pressure in cylinder 1

P_2 = Original pressure in cylinder 2 which is atmosphere pressure

P_f = Final pressure in the two cylinders when they are interconnected

V_1 = Volume of cylinder 1, fixed cylinder

V_2 = Volume of cylinder 2, test cylinder

V_m = Volume of material

V_c = Volume of calibration test block

Since:

$P_1V_1 = M_1RT$

$P_2 (V_2 - V_m) = M_2RT$,

and

$$P_f (V_1 + V_2 - V_m) = (M_1 + M_2) RT = P_1V_1 + P_2 (V_2 - V_m),$$

Then

$$V_m = \frac{P_f (V_1 + V_2) - P_1V_1 - P_2V_2.}{P_f - P_2}$$

Using manometer readings, rather than absolute pressures, yields:

$$V_m = \frac{(h_f + h_a)(V_1 + V_2) - (h_1 + h_a) V_1 - h_aV_2}{h_f + h_a - h_a}$$

or

$$V_m = \frac{h_f (V_1 + V_2) - h_1V_1}{h_f}$$

Volume Determination

Determining the original volumes, V_1 and V_2 can be done easily by constructing a small test cylinder having about half the volume of V_1. The procedure is as follows:

1. With the connecting valve closed between V_1 and V_2, pressurize V_1 and open V_2 to the amosphere.
2. Close the atmosphere on V_2 and close the air supply to V_1.
3. Read P_1.
4. Open the valve connecting the cylinders and read P_f.
5. Repeat steps 1 to 4 with the calibration block inserted in V_2, call the pressures obtained P'_1, P'_2, and P'_f.

Then

$P_f (V_1 + V_2) = P_1 V_1 + P_2 V_2$,

and $P'_f (V_1 + V_2 - V_c) = P'_1 V_1 + P'_2 (V_2 - V_c)$,

or $h_f (V_1 + V_2) = h_1 V_2$

and $h'_f (V_1 + V_2 - V_c) = h'_1 V_2$.

The two simultaneous equations can be solved for V_1 and V_2.

Some ingredients, particularly those containing oils, are difficult to sieve. It was found that the addition of about 0.4% of Cab-O-Sil, M.S.,* to the test material caused the particles

*Cab-O-Sil was made available by The Cabot Corp., Boston.

TABLE 1. Physical Properties of Some Common Microingredients and Ingredients

Ingredient	Density Gm./CC	F.M.	M.U.	D	S
AMINO ACIDS					
Lysine Supplement with wheat midds	1.28	1.42	0:1:9	226	2.07
L-Lysine Monohydrochloride	1.16	.09	0:0:10	66	1.51
Lysine 20% with wheat midds	1.32	1.36	0:1:9	209	2.28
Methionine DL-98%	1.17	.299	0:0:10	103	1.46
Methionine DL	1.16	.539	0:0:10	167	1.89
Methionine Hydroxy Analogue, 90%	1.37	1.42	0:3:7	200	3.15
ANTIBIOTICS					
Aureomycin *R diluted with soybean meal, soybean feed and fermentation solubles	1.38	1.95	0:4:6	325	2.51
Aureomycin with soybean meal and soybean feed	1.26	1.94	0:3:7	333	2.10
Aureomycin *R	1.27	1.70	0:3:7	296	2.14
Bacitracin *R	1.25	.81	0:1:9	141	2.31
Bacitracin, with diluents	1.33	.39	0:0:10	155	2.04
50% Penicillin	1.57	.44	0:0:10	110	1.66
50% Penicillin with oyster shell	1.52	.37	0:0:10	102	1.63
Procaine Penicillin G	1.22	.65	0:0:10	132	1.76
Streptomycin *R	1.47	.03	0:0:10	63	1.41
Terramycin *R	1.33	1.09	0:1:9	172	2.34
Tylosin *R gelatin beadlets	1.26	2.00	0:0:10	218	1.69
DRUGS					
ABC Ethylene	2.43	1.4	0:2:8	222	2.23
Ampral 20% with soy oil and corn gluten	1.21	2.54	0:1:9	258	1.69
100% Arsanilic Acid	1.52	.15	0:0:10	72	1.58
Arsanilic Acid with wheat midds	1.40	2.49	0:1:9	266	1.96
Arzene *R	1.77	.69	0:1:9	116	2.38
Bifuran	1.37	.89	0:0:10	145	2.02
Cadmium Wormer	1.47	1.59	0:0:10	265	1.65
Destrol	1.42	1.51	0:1:9	244	1.75
Dynafac Premix Armour	1.34	.58	0:0:10	121	1.88
Eddie Req.	2.46	1.70	0:2:8	283	2.04
Histocarb	2.38	.11	0:0:10	84	1.41
Histosep-S	1.79	.14	0:0:10	81	1.73
Histostat *R	1.94	1.39	0:0:10	227	1.80
Interstate Phenothiazine	1.34	.147	0:0:10	72	1.60
Moorman's Med. Red Ery	2.10	1.61	0:2:8	256	2.39
NR-100 *R	1.35	1.84	0:0:10	317	1.36
NFZ *R	1.47	1.05	0:0:10	163	2.17
NFZ in citrus meal	1.35	1.93	0:3:7	333	2.05
NFZ *R Mix	1.49	1.04	0:0:10	158	2.29
Neomix	1.67	.23	0:0:10	83	1.65
3-Nitro *R	2.10	.048	0:0:10	62	1.39
Sulfa Veterinary	2.34	.02	0:0:10	64	1.33
Sulfaquinoxaline 40%	1.25	1.34	0:0:10	223	1.61
Stilbosol	1.36	2.27	0:1:9	427	1.74
Stilbestrol	1.35	2.0	0:3:7	363	1.70
TSC-80 Mad.	1.53	.52	0:0:10	117	1.72
Tristat	1.79	.05	0:0:10	69	1.42
Trithiadol Coccidiostat	1.81	.03	0:0:10	65	1.31
Trolene FM	1.87	2.19	0:2:8	405	1.36
Triverm Powder	1.43	.67	0:1:9	109	2.18
Unistat *R	1.74	.41	0:0:10	109	1.66
Whitsyn 10	2.64	.63	0:0:10	136	1.45
MINERALS					
Calcium	2.42	.03	0:0:10	63	1.41
Defluorinated Phosphate	2.95	1.69	0:4:6	223	3.33
Dicalcium Phosphate	2.35	1.62	0:2:8	288	2.06
Granular Feeding Calcium	2.59	1.59	0:3:7	255	2.31
Soft Phosphate	2.59	.969	0:1:9	143	2.55
Trace Mineral Premix	3.34	1.77	0:0:10	146	1.60
Trace Mineral Premix	3.15	.06	0:0:10	59	1.47
VITAMINS					
Vitamin A, 250,000 U.S.P./gm.	1.00	.559	0:0:10	123	1.65
Vitamin A, 250,000 U.S.P./gm.	1.14	1.40	0:0:10	234	1.49
Vitamin A, 325,000 U.S.P./gm.	1.26	1.29	0:0:10	209	1.42
Vitamin A, 325,000 units/gm.	1.25	1.71	0:0:10	289	1.41
Vitamin A, 10,000 U.S.P./gm.	1.28	1.41	0:0:10	227	1.66
Vitamin A, 325,000 U.S.P./gm.	1.26	1.47	0:0:10	245	1.43
Vitamin A, 100,000 U.S.P./gm.	1.20	1.73	0:2:8	290	2.03
Vitamin A, 30,000 U.S.P./gm.	1.10	2.26	0:4:6	427	1.78
Vitamin A/D_3, 325 units/gm.	1.26	1.40	0:0:10	234	1.43
Vitamin B_{12}, 24 mg. per pound	1.63	.80	0:1:9	137	2.17
Vitamin B_{12}	1.42	1.22	0:1:9	202	2.08
Vitamin B_{12}, 24 mg. per pound	1.38	1.60	0:2:8	264	2.03
Vitamin B	1.25	1.65	0:1:9	273	1.92
Vitamin C	1.55	.15	0:0:10	96	1.63
Vitamin D, 200,000 U.S.P./gm.	1.27	1.09	0:0:10	328	1.25
Vitamin D_2, 400,000 U.S.P./gm.	1.29	2.09	0:1:9	367	1.22
Vitamin D_3, 30,000 I.C.U./gm.	1.05	2.06	0:1:9	366	1.30
Vitamin D_3, 200,000 I.C.U./gm.	1.25	.61	0:0:10	131	1.77
Vitamin D_3, 200,000 I.C.U./gm.	1.24	1.69	0:0:10	377	1.34
Vitamin E, 20,000 I.U., equiv. to 20 gm./lb.	1.25	1.33	0:1:9	260	1.83
Vitamin E, 50,000 I.U./lb.	1.23	1.24	0:0:10	209	1.63
Vitamin E, 20,000 I.U./lb.	1.26	2.35	0:4:6	461	1.69
Vitamin E, 20,000 I.U./lb.	1.08	1.21	0:0:10	203	1.85
Vitamin E	1.17	1.29	0:0:10	217	1.40
Vitamin E	1.23	1.36	0:0:10	228	1.51
Vitamin K	1.30	1.62	0:2:8	252	2.00
Vitamin K, Menadione U.S.P.	1.13	(Difficult to sieve)			
Vitamin K	2.51	.079	0:0:10	73	1.35
Calcium Pantothenate	1.31	.34	0:0:10	92	1.76
Calcium Pantothenate	1.25	1.48	0:0:10	236	1.07
Calcium Pantothenate	1.29	2.17	0:4:6	398	1.79
Choline Chloride 27½% on cereal carrier	1.25	2.50	0:6:4	502	1.62
Choline Chloride 70% liquid	1.11				
Choline Chloride 37½% on cereal carrier	1.12	2.59	0:7:3	633	1.58
Choline Chloride 25% and soybean mill feed	1.20	1.52	0:1:9	246	1.94
Choline Chloride 25%	1.21	2.08	0:3:7	371	1.71
Choline Chloride 25% on wheat midds	1.21	2.36	0:4:6	326	1.95
Choline Chloride 50% on corn cobs and wheat midds	1.17	1.71	0:2:8	292	1.80
Choline Pantothenate 64 gm./lb., diluted with calcium carbonate and rye middlings	1.47	.77	0:0:10	141	2.01
Niacin, 99% pure	1.29	.29	0:0:10	102	1.49
Niacin, 50% diluted with wheat midds	1.26	1.49	0:0:10	247	1.74
Pyridoxine Hydrochloride U.S.P. (Vitamin B_6)	1.26	.03	0:0:10	69	1.30
Riboflavin 98.1% pure	1.18	1.28	0:0:10	215	1.47
Riboflavin w/various diluents	1.76	1.15	0:1:9	180	2.14
Riboflavin w/diluents	1.22	1.40	0:2:8	232	2.05
Riboflavin	1.28	1.67	0:0:10	201	1.43
Riboflavin, 96% pure	1.27	1.61	0:0:10	252	1.45
Thiamine Hydrochloride U.S.P.	1.31	2.20	0:4:6	405	1.76
OTHER INGREDIENTS					
Corn	1.32				
Corn Distillers Solubles	1.12	.58	0:1:9	119	2.07
Distillers Solubles	1.15	1.32	0:1:9	220	1.89
Dried Beet Pulp	1.05	3.88	0:10:0	1299	1.27
Dried Whey	1.42	.54	0:0:10	110	2.01
Ethoxyquin on oat mill feed and calcium silicate	1.23	.28	0:0:10	88	1.61
Ethoxyquin on vermiculite	.80	1.70	0:0:10	503	1.42
Meat Scraps	1.37	1.67	0:3:7	261	2.18
Oats	1.35				
Salt	2.20				
Soybean Oil Meal, solvent	1.26	1.64	0:0:10	274	1.61
Sorghum, grain	1.35				
Urea	1.29	1.70	0:1:9	296	1.63
Urea Feed	1.26	1.47	0:0:10	243	1.66

F.M. = Fineness Modulus.
M.U. = Modulus of Uniformity.
D = Geometric mean diameter, microns.
S = Geometric standard deviation.
*R = Trade name of product.

to separate and pass through the sieve openings readily.

Table 1 shows the particle size and density of the feed ingredients tested, using sieve analysis; Table 2 shows the particle size and density of ingredients measured with a microscope.

Conclusions

From a review of the properties shown, several conclusions can be made. It is apparent that most of the vitamins are about similar in density and size to ground grains and should not be particularly difficult to mix. Some minerals are quite fine and dense and should be a critical factor in mixing. Some of the drugs are intermediate in density and may be more difficult to mix than the vitamins but probably less so than minerals.

A few ingredients are quite small and could easily be lost from a feed mixture in some processes, such as those using cyclones, unless they are made to adhere to a larger carrier particle.

REFERENCES

1. Bloom, C., and Livesey, E. F.—Particle Size for Additives to Animal Feeds! Chemical, 24 371-373, September, 1953.
2. Brueggemann, J., and Niesar, K. H.—Der Einfluss der Morphologischen Beschaffenheit der Mischkomponenten auf den Homogenisierungsgrad eines Mischung. Dechema-Monographien der Verfahrenstechnik, Hamburg, Germany.
3. 1966 Agricultural Engineers Yearbook - Methods of Determining Modulus Uniformity and Modulus of Fineness of Ground Feed. The American Society of Agricultural Engineers, 17th edition, November, 1949, revised 1962, p. 279.
4. 1961 Feed Production Handbook - Methods of Determining Modulus Uniformity and Modulus of Fineness of Ground Feed. The American Society of Agricultural Engineers, St. Joseph, Mich., November, 1949, p. 28.
5. Bradley, V. E., and Pfost, H. B.—Describing Particle Size Distribution of Feedstuffs Statistically; Feedstuffs, Nov. 5, 1966, p. 58.

ACKNOWLEDGMENT

The authors wish to acknowledge the extensive financial support of the Prater Pulverizer Co. which made this research possible.

Contributors of ingredients include: Armour and Co.; Atomic Basic Chemical Corp.; American Cyanamid Co.; Calcium Carbonate Co.; Commercial Solvents Corporation; Dawe's Laboratories, Inc.; Distillation Products Industries; Dow Chemical Co.; Eli Lilly and Co.; Glend-O-Lac Co.; Hess and Clark; Hoffmann-La Roche Inc.; Hoffmann-Taff, Inc.; Interstate Chemical Corp.; Merck and Co., Inc.; Monsanto Chemical Co.; Rhodanese Mfg. Co.; Michigan Agricultural Co.; Rogco Chemical Co.; Chas. Pfizer & Co.; Rohdeen Laboratories; Smith Douglass Co.; Sterwin Chemicals Inc. and Willmoyer, Laboratories, Inc.

TABLE 2. Microscopic Particle Size Analysis

Ingredient	Average Measurement (in microns)	Density gm./CC
Amprol *R	7.8	1.22
Ascorbic Acid	15.6	1.65
Cobalamin Concentrate	2.8	1.80
Hepalde *R	12.0	.993
Menadione	21.8	1.13
Nicarb *R	7.5	1.51
Riboflavin 98.1% pure	15.0x2.2	1.10
Riboflavin 95% pure	22.2x2.4	1.27
Sulfaquinoxaline	13.4	1.27
Thiamine	23.8	1.31
Trace Minerals	19.6	3.34
Vitamin D_3	94.4	1.29
Vitamin D_3	74.0x45.2	1.27

*R Trade name of product.

Reprinted from FEEDSTUFFS *of November 12, 1966*
(Vol. 38, No. 45, p. 62)

PART 4—Feed Mixing Series

Testing Feed Mixtures, Mixers And Related Equipment

By Dr. Harry B. Pfost, Dr. Charles Deyoe, Carl Stevens and Edward Morgan

Kansas State University

Previous articles of this series (1, 2) have discussed statistical concepts of evaluating feed mixtures and the physical properties of the most commonly used macroingredients and microingredients.

Relatively little literature is available regarding methods suitable for testing the performance of feed mixers and related conveying and storage equipment. Wornick (3) reviewed the literature before 1956. Hastings et al. (4) reported tests conducted with a small premix type mixer. Creger (5) reported on some factors that affect mixing and the effect of vitamin A variations on chick growth. Bruggemann and Niesar (6, 7) have reported on extensive testing of various types of mixers commonly used in Europe but their work is relatively limited in regard to assay methods reported. The American Institute of Chemical Engineers (8) has proposed standard testing procedures for mixers but did not attempt to prescribe assay procedures. The Merck Service Bulletin (9) gives many practical suggestions on testing.

Ideally a feed mixture should have all ingredients uniformly distributed throughout the mass of material so the animal receives a uniform amount of each critical nutrient each day. Since the nutritional requirements of the animal must be supplied, the accuracy of mixing should be related to animal needs. A baby chick consumes approximately 13 gm. of feed per day during the first 10-14 days of its life; hence, magnitude of samples of feed for chicks should be about 15 gm. Samples for laying birds might be about 115 gm. Samples of swine feed might range from about 30 gm. of a pig starter to 3,000 gm. of a swine finishing ration. Large ruminant animals consume larger amounts so samples of 10,000 to 20,000 gm. might be taken in dairy or beef finishing rations.

EDITOR'S NOTE: The authors of this article, the fourth in a five-part series, are: Dr. Pfost, professor; Dr. Deyoe, associate professor; Mr. Stevens, former agricultural extension specialist, and Mr. Morgan, former research assistant, department of flour and feed milling industries, Kansas State University, Manhattan, Kansas. The article constitutes contribution No. 222 of the Kansas Agricultural Experiment Station.

Fat soluble vitamins, A, D, E and K, are generally stored in the body for varying periods and the day-to-day variation is probably not critical. On the other hand, water-soluble B-complex vitamins probably are required at optimum levels each day.

Effect of Delay

Studies with rats (10, 11, 12) have shown that a delay of one hour or more in supplying lysine, in lysine deficient diets, results in poor performance of the animal and poor utilization of lysine. The average food passage time for growing chicks and high producing hens is approximately three to four hours. Thus, a delay of three hours or more in supplying an essential amino acid could severely reduce the value of the protein supplied.

Some materials are beneficial at low levels and may be toxic at higher levels. Urea may be fed safely at about one fourth pound per day to mature dairy or beef animals but one pound per day is near the toxic range. Considering the possibility of one animal consuming a particularly large amount of feed in a single day, there is a relatively small factor of safety for poor mixing of urea.

Salt is a common ingredient but large excesses with low water intake may retard growth or cause death.

Studies with some of the common coccidiostats, blackhead agents, etc., have shown that excesses of four or five times recommended levels can result in significant reductions in performance. Candren et al. (13) found that excessive levels of the drug 1, 2-dimethyl-5-nitroimidazole severely reduced growth and increased mortality of turkeys, Table 1.

Pfost (1) has described the effect of the Poisson Distribution due to a limited number of particles in a sample. To keep the error due to this effect below 3%, at least 900 particles of the material being assayed for must be included in the sample. Sometimes it may be impossible to secure a sample that approximates the animal's daily ration and provide sufficient particles. For example, because of the low level of vitamin D, which is required by baby chicks, one would seldom find 900 vitamin D particles in a 15 gm. sample. Hence, the selection of sample size or the interpretation of assay results must consider sample size and number of particles expected.

If an extremely large sample must be taken to secure an adequate number of particles and such a sample is too large for the assay to be used, then the entire sample may be ground and divided to secure an adequate number of particles in a small sample. The authors used this procedure in testing a mixture of a pelleted supplement in rolled barley.

The number of samples to be taken depends on the accuracy of the results desired. Cahn et al. (14) discuss the problem of selecting the number

TABLE 1. Effect of Level of a Nitro Compound on Mortality (%) of Turkeys

	Time (weeks)						
	(0-4)	(4-8)	(8-12)	(12-16)	(16-20)	(20-24)	(0-24)
Control	3	2	3	1	0	2	11
0.10% Nitro Compound	1	3	1	0	3	3	11
0.20% Nitro Compound	5	52	30	1	1	0	89

Effect of Level of a Nitro Compound on Growth (lb.) of Turkeys

	Time			
	4 weeks	8 weeks	12 weeks	16 weeks
Basal	1.57	4.23	8.86	12.4
0.10% Nitro Compound	1.54	4.07	8.20	11.7
0.20% Nitro Compound	1.37	3.22	5.48	9.2

FIGURE 1. Separatory Funnel Used for Sedimentation Assays

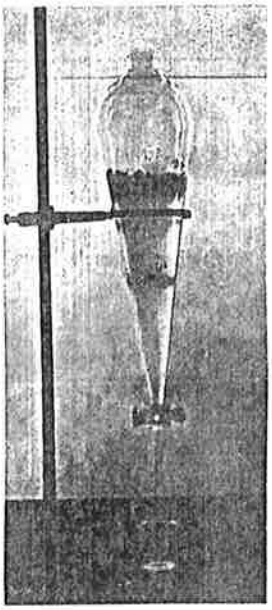


FIGURE 2. Relationship of Sedimentation to Chloride Analysis

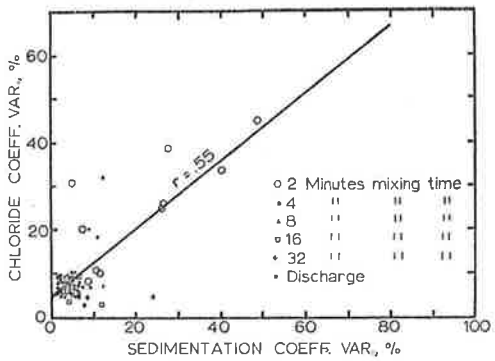


FIGURE 3. Relationship of Sedimentation to Chloride Analysis

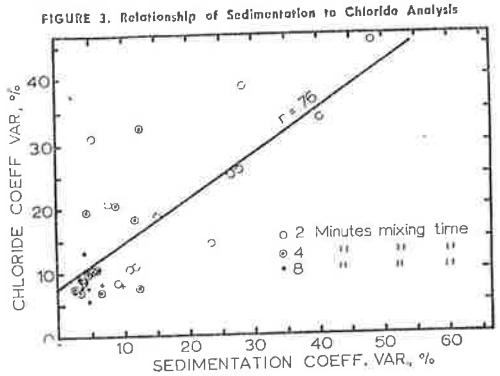


FIGURE 4. Relationship of Sedimentation to Kjeldahl Analysis

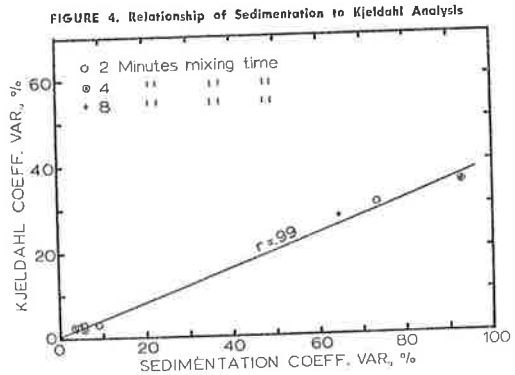


FIGURE 5. Relationship of Sedimentation to Amprol Analysis

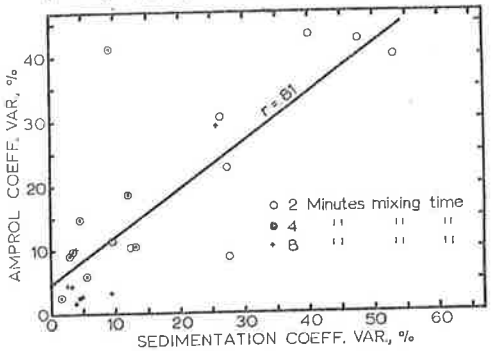
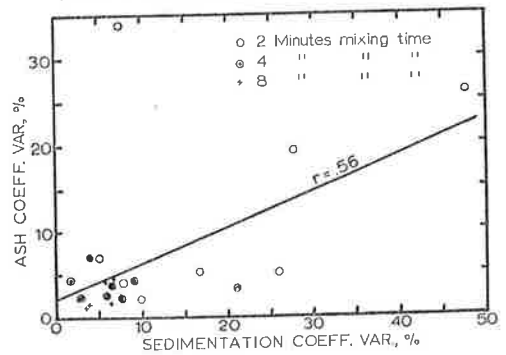


FIGURE 6. Relationship of Sedimentation to Ash Analysis



of samples required; they selected eight samples as being appropriate for their research on mixing chemicals in a V-mixer. Bruggemann and Niesar generally used 10 samples from a mixer. The authors, in research to be reported later, took 12 samples from within the mixer and/or 10 samples from the mixer discharge or sack-off bin discharge. For quality control, as opposed to research, the number of samples might be reduced to five without seriously reducing reliability of the results.

Cahn et al. (14) based their selection of the number of samples to be taken on a (1-α) confidence region for σ^2 so the probability of

$$\frac{(S-1)\sigma n^2}{X^2\,(s-1,\ 1-\alpha/2)} < \sigma^2 < \frac{(S-1)\sigma n^2}{X^2\,(s-1,\ \alpha/2)}$$

S = number of samples to be taken after N revolutions of the mixer

σn^2 = sample variance after N revolutions

$X^2(s-1)$ = Chi square random variable on (s-1) degrees of freedom.

Taking a sample is not difficult if the final product is being sampled. If a stream of material can be completely interrupted by a container, the sample obtained should provide a representative sample at a particular time interval. The size of such a sample is usually larger than required and will have to be divided.

Samples of material may be scooped from unsewn bags. If the scoop is used in such a way as to disturb the mixture very little, it should provide a representative sample. Such samples frequently may be about the correct size for the assay.

If sewn bags, mixers, bins or tanks are to be sampled, then a probe or sampling thief should be used to withdraw the sample. The probe should be inserted with the holes covered. When the probe has been filled at the location to be sampled, the openings should be covered before it is removed. Probes may segregate some mixtures so they should be used carefully.

If representative samples have been taken, the mean assay values of any component for all samples should be very close to the expected value.

Ideally, samples should be taken at random from a lot of material. This is frequently difficult because of time, space or other limitations. Frequently, if a mixer discharge is being sampled, it is necessary to sample as rapidly as possible at almost uniform time intervals, which may mean taking samples at one to five second intervals. If a mixer is being probed, it may be difficult or undesirable to make the number of openings required in the mixer shell to give entirely random sampling throughout the entire volume of the mixer. If poor mixing or segregation is indicated after preliminary trials, it may be desirable to make a special effort to sample from particular locations or at particular times to locate trouble spots.

Sampling within a mixer may be particularly desirable under two conditions:

a. When a mixer is being studied, an attempt is made to determine where certain ingredients may concentrate.
b. When the effect of time of mixing is being studied and it is not desirable to discharge the mixer frequently or before mixing is complete.

When the rate of mixing is unknown and the form of the curve relating degree of mixing to time is being studied, internal sampling is particularly useful. For example, if a mixer required six minutes to yield an acceptable mix, this point of time might be determined best by internal sampling at 2, 4, 8 and 16 minute intervals. The results obtained would then indicate the approximate mixing time without the disadvantage of having produced poorly mixed material as would be the case if the mixer had been emptied and sampled after a 4-minute mixing period.

TABLE 2. Recovery of Salt and Limestone by Sedimentation Assay from Individually Prepared 30 gm. Samples of Soybean Meal and Minerals

1% Salt Added		1% Salt and 1% Limestone Added	
Sample No.	Sediment %	Sample No.	Sediment %
1	1.14	11	1.70
2	1.10	12	1.79
3	1.13	13	1.72
4	1.11	14	1.73
5	1.15	15	1.75
6	1.11	16	1.76
7	1.15	17	1.76
8	1.14	18	1.75
9	1.15	19	1.75
10	1.18	20	1.73
$\bar{X}$ = 1.136		$\bar{X}$ = 1.74	
S = .0238		S = .025	
C.V. = 2.10%		C.V. = 1.42%	

TABLE 3. Recovery Data from Prepared Samples of Ground Sorghum Grain and Sodium Chloride Using the Potentiometric Assay for Chloride Ion

Sample No.	Na Cl Added %	Na Cl Found %	Recovery %
1*	0	.07	100
2	.25	.33	103
3	.50	.49	86
4	.75	.81	99
5	1.00	1.11	104
6	1.25	1.22	92

*Replicated.

TABLE 4. Assay Variation of Potentiometric Chloride Determination of Sodium Chloride. Samples Were Taken from a Single Reground Sample of Rolled Barley and Pelleted Supplement

Sample No.	Na Cl, %	Sample No.	Na Cl, %
1	.361	6	.383
2	.402	7	.375
3	.350	8	.405
4	.376	9	.390
5	.302	10	.406

X = .383
S = .0102
C.V. = 4.76%

TABLE 5. Coefficients of Correlation, r, Between Various Assays at 2, 4 and 8 Minute Mixing Times in a Vertical Mixer

Assay	r
Amprolium compared with Chloride	.92
Sedimentation compared with Amprolium	.81
Sedimentation compared with Chloride...	.76
Sedimentation compared with Ash	.56

Assays

Each sample taken to determine the degree of mixing should be assayed for each critical nutrient element, drug, etc. However, the cost of such assays may make it desirable to restrict attention to certain critical elements. Some of the factors to be considered in selecting tracers include:

a. There is little to be gained from being unduly concerned about assays for elements where variation would not affect animal performance, e.g. vitamin A.
b. Ingredients with almost identical physical properties need little attention. For example, soybean meal and ground corn have almost the same density and particle size; such ingredients should mix well under any conditions.
c. If practically all of the ingredients in a mixture have the same characteristics in some respect, do not attempt to assay for that characteristic. For example, oats, corn and soybean meal have relatively the same ash content. Little can be learned from ash assays of a mixture of those three ingredients.
d. If the analytical method for a particular ingredient has greater variability than the true variation of a mixer, assay for that ingredient is not a suitable test of efficiency of the mixer.
e. Drugs can make good tracers because the degree of mixing is important from both a legal and animal performance standpoint. Further, accurate assays are available for most drugs and generally there is only one ingredient source for the drug. Also as shown by Pfost, et al. (2), many drugs have relatively small particle size and high density.

PART 5—Feed Mixing Series

Testing and Performance Of a Vertical Twin Screw Mixer

By Dr. Harry B. Pfost, Dr. Charles W. Deyoe, Edward Morgan, Carl Stevens and Dr. Roshan Chaddha

Kansas State University

EDITOR'S NOTE: This article, the concluding one of a five-part series, was prepared by the following: Dr. Pfost, professor; Dr. Deyoe, associate professor; Mr. Morgan, former graduate research assistant, and Mr. Stevens, former research assistant, department of flour and feed milling industries, and Dr. Chaddha, former assistant professor, department of statistics, Kansas State University, Manhattan, Kans. The article constitutes contribution No. 511 of the department of flour and feed milling industries and No. 107 of the department of statistics, Kansas Agricultural Experiment Station.

Previous articles of this series (Parts 3 and 4) discussed problems in ...ating and testing feed mixers. Little literature is available regarding techniques for testing performance of animal feed mixers or for determining mixing time required by commercial mixers under practical operating conditions. Bruggemann and Niesar (4, 5) have reported results of tests conducted on several types of feed mixers commonly used in Europe.

FIGURE 1. Photograph of Test Mixer

The performance of a feed mixer depends on the design of the mixer and physical properties of ingredients to be mixed. A mixer may perform satisfactorily but quality control may be lost by segregation during handling and storage so a poorly mixed feed leaves the mill.

Feed manufacturers also must be concerned with possible contamination by inadequate discharge or incomplete cleaning of the mixer.

These studies were initiated to determine several factors related to the performance of a specific commercial vertical mixer. Factors thought to be of primary importance included:

a. Required mixing time.

b. Satisfactory mix of ingredients of diverse physical characteristics.

c. Effect of using various dilutions of premix.

Tested was a 2 ton, top loading, twin screw vertical mixer manufactured by the Prater Pulverizer Company (Figure 1). Each screw was powered by a 7½ hp. electric motor. The average speed of each screw was 292 rpm. on all but one series of tests. The working capacity of 151 cu. ft. was adequate for 2 ton batches of most complete feeds but the size of a batch for rations containing large amounts of rolled or ground oats or barley was reduced to 1½ tons.

Figure 2 is a schematic view of the mixer showing locations of four holes made to allow internal sampling of loads. Samples were taken internally at specific times, three from each hole with a grain probe. Figure 3 shows the flow from the mixer to the 6 ft. diameter sack off bin. Assay methods used were described in reference 3.

FIGURE 2. Schematic View of Mixer Showing Probe Locations

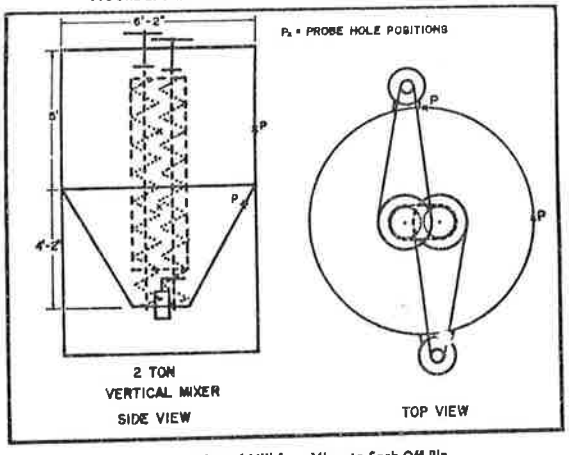
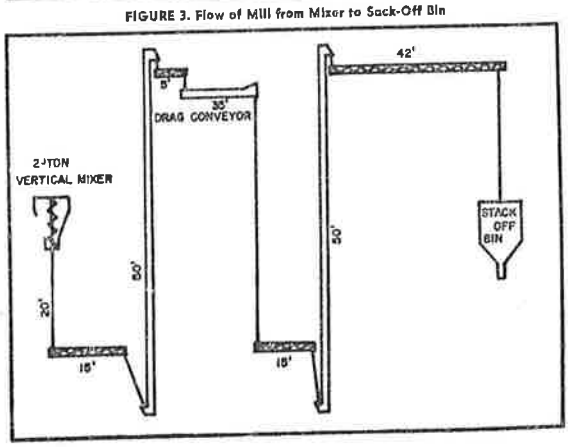


FIGURE 3. Flow of Mill from Mixer to Sack-Off Bin



Studies Mixing Time

Time required for feed material to make a complete cycle in the mixer was determined by filling it with a 2 ton batch of a complete poultry feed, with colored salt tracer particles on top; red particles near the outside, and blue particles near the screw housing. Samples of the discharge from the screws were taken at five second intervals. The results indicated a 35 second cycle for material moving down the center of the mixer and a 40 second cycle for material moving down along the sides of the mixer.

Time a mixer should be operated might be determined on the basis of two criteria:

a. The time required to achieve a satisfactory mix might be used as a guide. Reference 1 indicates that a coefficient of variation of 5 to 10% is probably satisfactory in most cases to meet reasonable nutritional and regulatory requirements. That is called a "satisfactory mix" here.

b. Time required to reach a minimum variation where any additional mixing time will not cause a significant ($P < .05$) change in sample to sample variation. Called "best mix" here.

Mixing time definitions depend on how ingredients are added and when the mix is considered to have started. In tests reported here, all major ingredients were placed in the mixer with the mixer stopped. Tracer materials, usually in an undiluted form, were then placed on top the charge in the mixer. Mixing time started when the mixer was turned on. Mixing time would have been shorter had the test material been added with the mixer operating and the mixing time started when the last of the material was in the mixer. Mixing time for this mixer also is reduced if the tracer material

TABLE 1. F-test for Variance of Data Obtained When Mixing a Chick Grower Ration Indicated Time Intervals

Assay	Treatments	P	Table F	Calc. F
Sed.	4 minutes vs. 6 minutes	0.05	1.70	1.38*
Sed.	6 minutes vs. 8 minutes	0.05	1.75	1.52*
Chloride	4 minutes vs. 6 minutes	0.05	1.72	1.60*
Chloride	6 minutes vs. 8 minutes	0.05	1.92	1.02*
Amprolium	4 minutes vs. 8 minutes	0.05	1.78	1.20*

*Significant at P = 0.05.

TABLE 2. Effect of Pelleting a Supplement Before Mixing with Steam Rolled Barley

Type of Supplement	Type of Assay	—Coefficient of Variation, %*— Sampled at Discharge	Sampled at Bag
Mash	Sedimentation	46.6	54.0
Mash	Chloride Ion	33.8	53.4
3/16 In. Pellet	Chloride Ion	8.4	14.6

*Average value of three replications.

TABLE 3. Analysis of Variance of Mixing Tests; Ground Corn and Salt Mixed; Sedimentation Assays

Source of Variation	DF	SS	MS	F
Location L[1]	3	.05699	.01899	.02146
Samples S[2]	2	.003164	.001582	.1787
Time T	4	.00225	.02056	.02322
L X S	6	.01402	.002337	.2640
L X T	12	.09641	.008034	.9076
S X T	8	.03977	.004971	.5616
Error	24	.2124	.008851	
Total	59	.5050		

[1]Indicates probe hole location.
[2]Indicates sample location along probe.

TABLE 4. Analysis of Variance of Mixing Tests; Rolled Corn and Salt Was Mixed and Sedimentation Assays Were Made

Source of Variation	DF	SS	MS	F
Location L[1]	3	.2051	.06837	.03034*
Samples S[2]	2	.1684	.08422	.03737*
Time T	4	.2629	.06573	.02917*
L X S	6	.5876	.09793	.04346**
L X T	12	.04414	.3678	.001632**
S X T	8	.2530	.03163	.01404 NS
Error	24	.5407	.02253	
Total	59	.06432		

*Significant at P≥0.05.
**Significant at P≥0.01.
[1]Indicates probe hole location.
[2]Indicates sample location along probe.

the top of the mixer as in the earlier tests. In all cases typical chick grower ration was used. Figures 16 and 17 show results of the series. As expected, more dilute premixes, type 1, can be mixed in less time. It appears that a satisfactory mix of supplements with ground grain easily can be achieved in four minutes or less in the mixer tested. There was some indication that severe overmixing, as for 32 minutes, may be detrimental.

Analysis of Variance

The sampling technique used provided an opportunity to study, statistically, some factors that may affect a mixer's performance. Such an analysis might provide clues for improvements in mixer design. Data from a large number of tests were analyzed by computer. Results of two typical tests are given in Tables 3 and 4.

The analysis of variance table shows that for material that mixed well, ground corn, the only significant variable was time. For material that mixed poorly, rolled corn, location at which samples were taken gave significant differences. Analyses of such data may show that design features of a mixer need to be modified to improve its performance; however, in this case, a better solution is simply to obtain more equal particle sizes of the various ingredients to be mixed.

Conclusions

The results of the tests show that the mixer tested will provide a satisfactory mix in approximately four minutes, depending on type of ingredients mixed. No more than four minutes should be required when a typical supplement is mixed with ground grains. Undiluted microingredients will require slightly longer mixing times.

Certain combinations of types of ingredients are difficult or impossible to mix satisfactorily. Because of segregation during handling after mixing, mixing such combinations should be avoided. Ingredients highly diverse in particle size are difficult to mix and segregate easily on further handling. Steps that might reduce difficulties of mixing and handling ingredients of peculiar particle shapes or widely varying sizes include:

1. Grind major ingredients so their size is near the particle size of the critical ingredients, like drugs.
2. If the type of formula precludes grinding the major ingredients to a small size (as in rolled barley or corncob meal rations), then consider using a pelleted or crumbled supplement.
3. Mixing particles of diverse physical characteristics can be facilitated by adding liquids, like fats or molasses, at relatively low levels. Adding liquids will also help prevent segregation.

ACKNOWLEDGMENT

The authors sincerely appreciate the support of the Prater Pulverizer Co.

REFERENCES

1. Pfost, H. B.—Criteria for Evaluating Feed Mixer Performance; Feedstuffs, 36: 104-105, May 9, 1964.
2. Pfost, H. B., Charles Doyoe, Carl Stevens, and Edward Morgan — Physical Characteristics of Feed Microingredients; Feedstuffs, 38:45, Nov. 12, 1966, pp. 62-64.
3. Pfost, H. B., Charles Doyoe, Carl Stevens and Edward Morgan—Testing Feed Mixtures, Mixers and Related Equipment; Feedstuffs, 38:46, Nov. 19, 1966, pp. 32-33.
4. Bruggemann, J., and K. H. Niesar—Das Vermischungsproblem, Fachverband der Futtermittelindustrie ov. April, 1966.
5. Bruggemann, J., and K. H. Niesar—Der Einfluss der Morphologischen Beschaffenheit der Mischkomponenten auf der Homogenitatsgrad einer Mischung, Fachverband der Futtermittelindustrie ov., December, 1966.

FIGURE 14. Cumulative Logarithmic Screen Analysis on Samples of Unground and Ground Soybean Meal

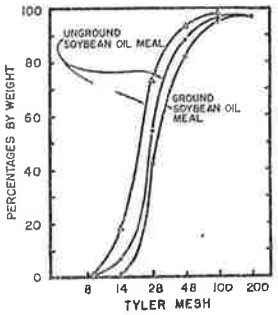


FIGURE 15. Cumulative Screen Analysis of Ground Limestone, Ground Soybean Meal and Salt

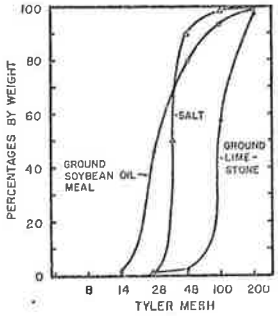


FIGURE 16. The Effect of Premix Size on Mixing

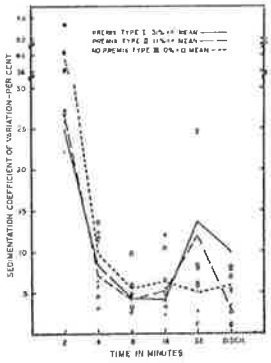
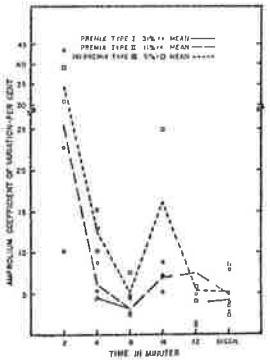


FIGURE 17. The Effect of Premix Size on Mixing



Reprinted from FEEDSTUFFS *of November 26, 1966*
(Vol. 38, No. 47, p. 62)

is diluted, as when contained in a supplement, as will be shown later.

Figure 4 shows the effect of mixing time with a typical complete turkey finishing ration. In this series both satisfactory and best mixes were obtained in four minutes.

Since the data in Figure 4, and data from other tests, indicated that the time between 4 and 8 minutes was critical, another series was made to determine more accurately the degree of mix achieved in the 4 to 8 minute range. The second series used a typical chick grower ration and two assay methods: sedimentation and chloride (described in reference 3). Figures 5 and 6 give the results. Questions regarding other nutrient elements may be answered by Figure 7, which shows results with other assays when a chick grower ration was mixed. Protein assay results show that major components mix quickly and are a poor measure of mixer efficiency.

To determine statistical differences among 4, and 6 and 8 minute mixing times (data in Figures 5, 6 and 7) an F-test for variances was made. The hypothesis tested is:

$$\sigma_1^2 = \sigma_2^2 \text{ at } P \leq .05.$$

Table 1 shows the results and indicates that mixing 6 or 8 minute does not improve results obtained in four minutes, based on sedimentation, chloride ion or amprolium assays.

Problem Ingredients

Since physical properties of ingredients were thought to influence mixer performance, exploratory tests were made to determine if the method of processing grains influences quality of the mix.

Sorghum grain, corn and oats were processed by grinding through a ¼ in. hammermill screen and by rolling with a 0.010 in. roller clearance. A mixture was made with salt as a tracer and sedimentation tests were used. Figures 8, 9 and 10 show the results. Rolled corn was too coarse to mix properly because of the extreme difference in particle size between corn and tracer material. The rolled sorghum grain was fine enough in this case to provide a satisfactory mix. Neither the ground nor rolled oats were suitable for mixing; apparently differences in particle shape, caused by oat hulls, prevented proper mixing.

The tests indicated that remedial measures must be taken to secure a proper mix of extremely diverse particle sizes and shapes. Since steam rolled barley is a more common feed ingredient than rolled oats, tests with barley were conducted to determine if a pelleted supplement would improve mixing characteristics. In the tests the mixer was charged with rolled barley and the supplement. Mash or $^5/_{16}$ in. diameter pellets were added at the top of the charge. After eight minutes of mixing, the mixer was discharged and samples were taken from the discharge stream and from bags after sacking off. When samples containing pellets were taken, a sample large enough (about 15 lb.) to include at least 1,000 pellets was taken. That provided a statistically reliable number of tracer particles (reference 1). The sample was ground before being divided to obtain assay samples to eliminate effects of Poisson distribution. The results (Table 2) indicate that a suitable sized pelleted supplement will dramatically improve mixing properties of formulas containing large amounts of steam rolled grains.

Frequently, in manufacturing supplements, a large fraction of the formula consists of soybean meal. Exploratory investigations indicated that particle size of soybean meal received at the university mill was too large to mix well with mineral ingredients. Figure 2 shows the results of typical mixing tests using soybean meal with ground limestone and the effect of adding 2% animal fat. Adding the fat clearly improved quality of the mix and reduced mixing time.

Since particle size had been shown to markedly affect mixing, grinding part or all of the soybean meal to improve mixing characteristics was tested. Ten and 20% of the soybean meal was reground through a ¼ in. hammermill screen. Regrinding a small fraction of the meal was not sufficient to give a good mix (Figure 12).

Mixing salt and limestone with soybean oil meal then was tested. Unground meal was compared with reground meal with 100% ground through a ¼ in. hammermill screen. Results are given in Figure 13. Regrinding the material improved the quality of the mix and reduced time required to obtain a satisfactory mix. Figures 14 and 15 show typical particle size data for ground and unground soybean meal, salt and limestone, and indicate that only a slight change in particle size of soybean meal may significantly change its mixing characteristics.

Effect of Premix Size

Tests reported above were made by filling the mixer with the major ingredients and then adding tracer material in a concentrated form at the top of the mixer. Since that is more rigorous than normal in practice, effect of diluting tracer material was tested. In the first case, a premix equal to approximately 31% of the total formula weight was used. That corresponds to common practice with a supplement and ground grain. In the second case, a premix equal to approximately 11% of the total formula weight was tested to correspond closely to using materials sometimes referred to as "super concentrates." In the third case, no dilution was made; tracer material simply was added at

FIGURE 4. Mixer Test Results Showing the Effect of Time When Mixing a Complete Turkey Finisher Ration

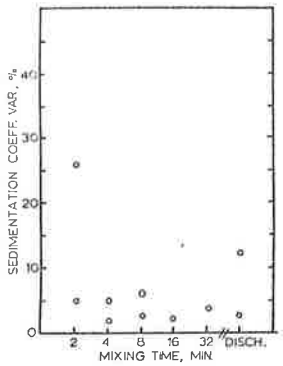


FIGURE 5. Mixer Test Results Obtained When Mixing a Complete Chick Grower Ration

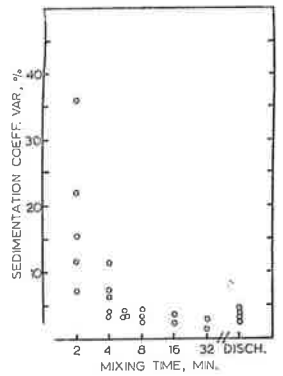


FIGURE 6. Mixer Test Results Obtained When Mixing a Complete Chick Grower Ration

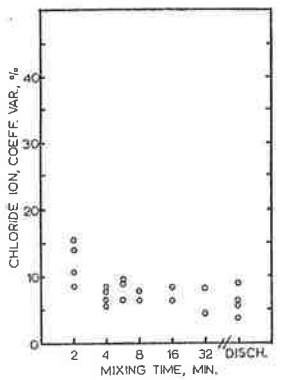


FIGURE 7. Comparison of Mixer Tests Using Drug and Protein Assays

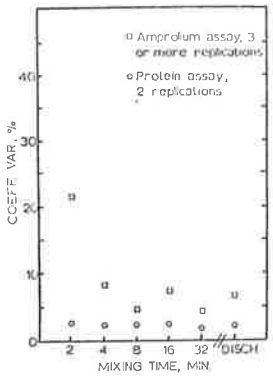


FIGURE 8. Effect of Grinding vs. Rolling of Sorghum Grain When Mixing with 1% Salt

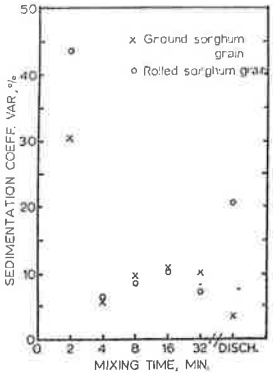


FIGURE 9. Effect of Grinding vs. Rolling of Corn When Mixing with 1% Salt

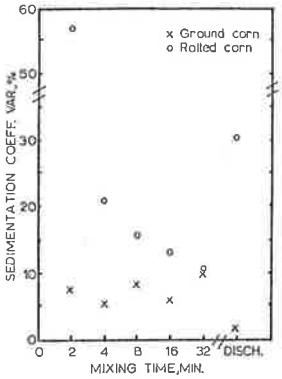


FIGURE 10. Effect of Grinding vs. Rolling of Oats When Mixing with 1% Salt

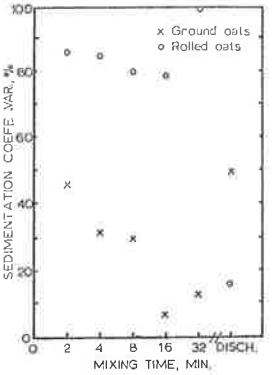


FIGURE 11. Effect of Adding Fat to Improve the Mixing of Soybean Meal and 5% Finely Ground Limestone

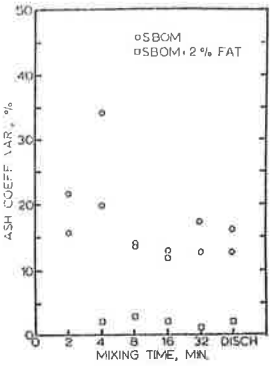


FIGURE 12. Effect of Grinding a Fraction of the Soybean Meal When Mixing with 1% Salt and 1% Limestone

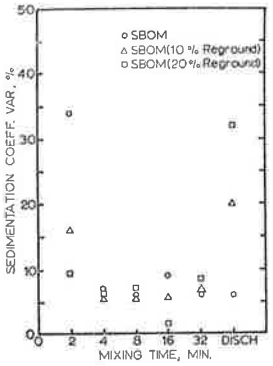
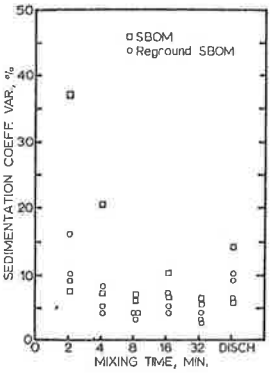


FIGURE 13. Effect of Grinding Soybean Meal Through a ¼ in. Hammer Mill Screen When Mixing with 1% Salt and 1% Limestone



f. Because of their high density and frequently small particle size, mineral elements may be good tracers. Their disadvantage may be relatively high assay cost and their presence in substantial amounts in many ingredients in a feed.

Pierce (15) has reviewed many of the assay methods that may be used to check mixer performance. He has not discussed drug assays but methods for them are readily available from drug manufacturers. Assay methods discussed here include only those used extensively by the authors in recent research on mixing at Kansas State University. Most of the methods were selected on the basis of their accuracy, low cost and ability to test a mixer under extreme conditions.

Colored Granule Method — Dyed salt tracer particles were investigated extensively. About 20 salt tracers per sample were all that could be adequately detected on a 4 in. filter paper. Since that number gives an assay coefficient of variation of about 22%, it was not used extensively. Using two colors of tracer particles as proposed by Midgley and Eisenberg (16) does not increase accuracy with a given total number of particles, but two colors may be useful for other reasons.

The ratio of the number of the two colors of particles found per sample is extremely difficult to handle statistically. The ratio of two Poisson distributions can be shown to be a Cauchy distribution; the well-known property of the Cauchy distribution is that it has no finite mean value.

Lanz et al. (17) have reported extensive use of colored granules for testing mixing and mixers. They have presented details of statistical analyses required to determine whether a mix gives a value significantly different from the value that might be expected from a perfect mix.

Vance (18) has reported on the use of particle counts in mixing and describes useful statistical tests which may be applied.

Ash Assays—These assays are relatively inexpensive and accurate. However, in normal feed mixtures, it was found that the test is poor for mixer evaluation because major feed ingredients contribute substantial amounts of ash to any sample and it is difficult to detect whether free minerals, which are present in low quantities, are mixed.

Sedimentation Tests — Figure 1 shows equipment used for sedimentation tests. Samples of about 30 gm. were used. Feed containing only 1% of minerals yields a substantial (0.3 gm.) amount of tracer, which may be separated readily from the cereal fraction.

A 1-liter separatory funnel was filled about two thirds full of a suitable liquid. Carbon tetrachloride is satisfactory but is somewhat hazardous and should not be used unless the work can be done under a chemical laboratory hood, where good ventilation is available. The authors have more recently used perchloroethylene because it is less volatile and hazardous. Any other liquid with low viscosity and specific gravity of over about 1.5 would be satisfactory.

The sample was placed in the funnel and stirred well to separate mineral particles from other particles. After a definite fixed time (we used five minutes) most of the mineral particles will settle to the bottom and the cereal and other light materials will float to the top of the liquid. Some very fine material particles will not settle in reasonable time, hence the necessity of using a fixed time interval.

The stop cock in the bottom of the funnel allows the mineral fraction to be separated into a beaker. After settling for a few minutes in a small, e.g. 50-100 cc. beaker, most of the liquid can be poured off the top and the liquid remaining on the mineral can be conveniently dried off in a drying oven (two hours) or under a ventilated hood in about 12 hours. The dried mineral is then weighed on a laboratory balance and converted to a fraction of the total original sample. This method easily detects the mix of the mineral fraction which would normally be thought to be difficult to mix because of its relatively high density and small particle size.

It was difficult to remove the finer minerals, e.g. limestone, from high moisture grain materials unless the assay was made within a short time, a few hours, after the sample was removed from the mixer. Fat levels of up to 2% added fat do not affect the accuracy of this method appreciably. Higher fat levels, or other liquids like molasses, have not been investigated. Table 2 shows recovery values for typical sedimentation assays.

Chloride Ion—Luhman (19) has reported a rapid method for potentiometric detection of soluble chlorides in feed. Since salt is a very common feed ingredient, dense, and obtainable in any desired particle size, it is a convenient tracer and one that probably is difficult to mix with most major feed ingredients. The potentiometric test is relatively inexpensive, rapid and requires only equipment that is available in most chemical laboratories. The authors extracted 10 gm. samples in 100 ml. of water. Tables 3 and 4 show the reproducibility of this method.

A relatively new chloride ion concentration indicator, sold under the trade name, Quantab®, can be used to detect the level of salt in a sample. The authors have had only limited experience with such indicators, but the manufacturers' specifications indicate that they are sufficiently accurate for many feed mixture tests. They do not require trained technical personnel and only a minimum of laboratory equipment, including balances that will weigh accurately within about 0.1 gm.

Amprolium—This assay was selected as a typical drug assay because it is sensitive, accurate, fairly easy and feed mixtures containing it were readily available for mixer tests. The material has about the same density as other feed ingredients and rather small particle size. It probably is not a particularly difficult material to mix and should represent rather average mixing properties.

During an extended period of mixer testing many assays were made using various assay methods described above. In an attempt to determine how well the various methods will predict the degree of mixing, correlations were made between various pairs of methods. Figure 2 shows a plot of coefficients of variation between sedimentation assays and chloride assays for a large number of assays taken from improperly mixed and well mixed materials. The correlation coefficient of all these data is quite low. Careful consideration of the problem led to the conclusion that the correlation coefficient would be more meaningful if the data from only improperly mixed material were considered. The original data represented a nonrandom sample from mixed and unmixed populations. Figure 3 shows the results of discarding all assays beyond eight minutes mixing time. Assays from well mixed populations naturally show a poor correlation because the coefficients of variations are due to random errors in sampling and analyses.

Figures 4, 5 and 6 show the relationships between other assay methods. Table 5 shows the coefficients of correlation between the various paired tests.

Conclusion

Several types of assays are available to test feed mixers and mixtures. Many are relatively inexpensive and rapid and can be used to test the degree of mixing of materials, like minerals, which because of their physical characteristics are probably difficult to mix or which tend to segregate.

For testing or research purposes, it is probably desirable to calculate deviations using about 10 samples.

For quality control purposes it may be possible to use fewer samples. The samples need not all be taken at the same time but may be taken singly, from time to time, and the results compiled if the level of the tracer remains constant throughout the period. This may allow a feed manufacturer to accumulate information regarding the efficacy of his mixing and handling process at the same time he is checking compliance with guaranteed levels of drugs, or other nutrient elements.

ACKNOWLEDGMENT

The authors appreciate the extensive financial assistance of Prator Pulverizer Co. in support of research reported here.

LITERATURE CITED

1. Pfost, H. B.—Criteria for Evaluating Feed Mixer Performance; Feedstuffs, 36:29, p. 104, May 9, 1964.
2. Pfost, H. B., Doyen, G. W., Stevens, C. A., Morgan, E. J.—Physical Characteristics of Feed Microingredients, Feedstuffs (submitted).
3. Wornick, Robert C.—Premixing Micro Ingredients, Lecture Series No. 4, Chas. Pfizer & Co., Inc.
4. Hastings, W. H., Feyerherm, A. M., and Kebkens, La Rue—Factors Affecting Microingredient Distribution in Feed Materials, Feedstuffs, 33:3, p. 38, Jan. 21, 1961.
5. Creger, C. R.—A Study of Distribution of Microingredients in Mixed Feeds, unpublished M.S. Thesis, Kansas State University, 1957.
6. Bruggemann, J., Nieser, K. H. — Das Vermischungsproblem, Fachverband der Futtermittelindustrie e. V., April 15, 1959.
7. Bruggemann, J., Nieser, K. H. — Der Einfluss der Morphologischen Beschaffenheit der Mischkomponenten auf den Homogenitatsgrad einer Mischung, Fachverband der Futtermittelindustrie e. V., December, 1962.
8. Solids Mixing Equipment, American Institute of Chemical Engineers, New York, N.Y.
9. Anonymous—Microingredients in Feeds, a Mixing Manual; Merck & Co., Inc., 1959
10. Geiger, E. — Experiments with Delayed Supplement of Incomplete Amino Acid Mixtures; J. Nutrition, 34:97-111 (1948).
11. Cannon, et al. — The Influence of Time of Ingestion of Essential Amino Acids on Utilization in Tissue Synthesis, Federal Proc., 6:320 (1947).
12. Henderson and Harris — Concurrent Feeding of Amino Acids; Federal Proc., 8:385 (1949).
13. Cauvren, H. B., Davies, R. E., Doyen, G. W., Zavala, M. A., Creger, C. R., and Couch, J. R.—Studies on the effect of 1,2-Dimethyl-5-Nitroimidazole on the growth and reproduction in turkeys and its residual concentration in tissue; Poultry Science, 42:588-604 (1963).
14. Cahn, D. E., Healy, T. W., and Fuerstenau, D. W.—Standar Geometry in the Mixing of Solids; I & EC Process Design and Development; Vol. 4, No. 3, July, 1965.
15. Pierce, James G.—Methods of Determination of the Distribution of Ingredients in Feed Mixtures; Feedstuffs, 33:36, p. 60 Sept. 5, 1961.
16. Midgley, M. G., and Eisenberg, Sylvan —How Efficient Is Your Mixer? Feedstuffs, 37:11, p. 62, March 13, 1965.
17. Lanz, G. T., Torchman, R., and Brew, W. B.—Evaluation of Mixing Tests; paper presented at 1965 Midwest Feed Manufacturers Assn., Kansas City, Mo.
18. Vance, Frank P.—Statistical Properties of Dry Blends, Ind. and Engr. Chem., 58:6, 37-44, June, 1966.
19. Luhman, G. A.—A Rapid Potentiometric Method for the Determination of Soluble Chlorine in Feeds; Association of Agr. Chem. J., 38:76, 4-756, Aug. 15, 1955.

Reprinted from FEEDSTUFFS *of November 19, 1966*
(Vol. 38, No. 46, p. 32)