MIXING STUDIES OF A VERTICAL MIXER AND SOME PROBLEM INGREDIENTS

by

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TABLE OF CONTENTS

INTRODUCTION

In 1962 the United States formula feed industry manufactured over 56 million tons of mixed feed for animal consumption (1). All, or practically all, of this feed was blended or mixed by some mechanical process in a machine called a mixer. For this reason and for others to follow, mixing can be called the heart of feed production.

"The Dictionary of Feed Terms" in the 1964 Feed Production School Transcript of Proceedings defines mixing as: "To combine by agitation two or more materials to a specific degree of dispersion." The importance of mixing is due in large part to the addition of small amounts of various drugs, vitamins and trace minerals to the feed ration. These additives must be distributed as homogeneously as possible, in accordance with the needs of the animal.

As Rumpf and Mueller (2) suggest, "The optimum result of a dry-solids mixing process is achieved when the single particles of the components are randomly distributed throughout the entire volume of the mix." This attempt at homogeneity through randomization is needed because of the possible effects on the animal of large variations of nutrients and additives. Feed manufacturers also place guarantees on feed composition and levels of additives contained in their feed, which are enforced by various governmental agencies.

In reference to variation in an animal's daily ration, Geiger (3) states that " . . . the nature of protein synthesis makes it mandatory to assume that the non-essential as well as the so-called essential amino acids must be available simultaneously

and in ample quantities to permit protein synthesis." Creger (4) used Vitamin A to simulate mixed and unmixed rations for young poultry. It is also well known that toxicities may result from large intakes of materials which are required only at low levels. Salt is one example of this: a large excess coupled with a low water intake can retard growth and even cause death (5), (6). Wornick (7) shows that the feed intake of a chick for the first week of life was approximately 70 grams. He states that "Such minute intakes demand uniform dispersion of all ingredients. The degree of mixing may also affect animals other than poultry. According to Deyoe (8):

> Baby pigs (10 lbs.) consume about .8 of a pound of feed per day . . . . Thus while the formula may contain adequate amounts of nutrients, poor mixing, combined with a low feed intake [as in the young animal] . . . could easily result in poor performance . . . . and typical deficiency symptoms and death in some cases.

These problems of poor distribution and/or segregation are probably due to the type of ration, problem ingredients, handling and mixing equipment, and duration of mixing.

Thus one can see the need in the formula feed industry, for a standard measure of mixing, and for rapid, inexpensive tests to determine the degree of mixing. The purpose of this research was to study, in a vertical mixer, the effects of time, a speed differential of the mixing screws, sizes of premixes, and ingredient characteristics on the homogeneity of mixing. Various assay methods were also investigated. This research was also undertaken in the hope of offering some solutions to special problems involved in mixing animal feeds.

REVIEW OF LITERATURE

The number of papers on mixing is rather voluminous and in most cases not particularly applicable to feed mixing. However, we shall attempt to touch on the more important works for information on theory and statistics.

Two names which are important in the compilation of mixing theory are Lacey and Weidenbaum. Lacey (9) states that "It is shown that there are three components in a mixing process: convection, diffusion and shear." He also examines other theories and compares them with published data. More prolific and far reaching is Weidenbaum, who in Advances in Chemical Engineering, Vol. II (10), summarizes a variety of past methods used to "determine the degree of mixing of a solid mixture . . . with short resumés of the papers from which they were taken." In a review of Gray's study of three solid systems consisting of sand-ilmenite, barium sulfate-ilmenite, and aluminum oxide-ilmenite, Weidenbaum (10) states that:

> The principal conclusion reached was that the rate of mixing obtained depended on the properties of the solids mixed as well as the type of equipment. When segregating tendencies were diminished, there were fewer differences among the various pieces of equipment. The method of loading the materials was shown to affect the rate of mixing in the ribbon mixer.

In a study of the mechanism of segregation in dry blended fertilizers, Pincan, et al, (11) mathematically developed a "friction factor" to predict segregation. Hoffmeister, et al, (12) investigating the effects of size, shape, and density on segregation report that:

> The tendency of dry blended fertilizers to segregate during handling and spreading was shown to result chiefly from differences in particle size of the various components of the blend [two materials] . . . . Variations in particle density had little effect, and shape had practically no effect . . . . Exploratory tests indicated that vibration (as occurs in bulk spreader trucks being driven to and across fields) was only a minor cause of segregation.

Smith's work (13) indicates that most mixtures can be transported 30 miles in a spreader truck with little segregation due to vibration.

The method of sampling and the size of the samples must not be overlooked due to their effect on test results. Kaufman (14) tested the dry mixing of antibiotics in a double-cone blender and three twin-shell blenders of different capacities. He believes it is important that "The sample size be directly related to the final product and sub-division. The number of samples be determined by the degree of confidence desired." In his work he used 10 random spot samples taken in the mixer after different numbers of blender revolutions. In working with soil, Smith (13) indicated that if the particle size of the major constituent is large, a large total sample must be taken to make the variance due to sample size small in comparison with the per cent additive use. Wornick (15) lists the factors affecting sample size as:

> (a) Level of addition of premix to the finished feed; (b) intended use of feed ; (c) batch size; (d) sample size required for testing; (e) potency of individual active ingredients incorporated; (f) particle size of active ingredients and carrier; (g) economics.

Poundstone (16) believes that "With . . . the necessity for thorough mixing of micro-ingredients, uniformity of mix is paramount and a portion of feed taken at any point in a bag or bin should be a satisfactory sample." He also suggests keeping the samples separate to check variability in the composition of the batch. In their experimental work, Blumberg and Maritz (17) conclude that 10 spot samples at each mixing stage were sufficient to plot degree of mixing versus time, which would accurately indicate when mixing was completed.

There have been several indexes proposed to measure mixing. Lacey (9) thinks that ". . . the most useful way of expressing degree of mixture is by measuring the statistical variation of composition among samples and using a mixing index, "M", defined as:

$$M = \left[\frac{S_o^2 - S^2}{S_o^2 - S_r^2}\right]$$

Where S_r^2=calculated variance of a random mixture, S_o^2 =calculated variance of the unmixed system, and S^2=variance of spot samples. M then goes from 0 to 1 as the variance goes from S_o (unmixed) to S_r (mixed).

In a mathematical treatment of the mixing problem based on chi-square using particle counts, Gayle, et al, in two publications (18), (19) proposed a segregation index:

$$S = \left[\frac{\chi_o^2 - \chi_r^2}{\chi_s^2 - \chi_r^2}\right]$$

Where χ_o^2 observed chi-square for any mixture, χ_r^2 expected chi-square for a random mixture, and χ_s^2 expected chi square for a segregated mixture. This index varies from one to zero, as mixing proceeds from segregation to randomization.

According to Hastings (21) "Perfect mixing is attained when the concentration of an additive in all possible samples equals the concentration in the entire batch: $C_i = C_o$ 'that is', the concentration in any observed sample equals the batch concentration." In this ideal case the standard deviation is equal to zero. However, since perfect mixing is not obtainable in practice the standard deviation can only approach zero. As a measure of mixing Hastings proposes the standard deviation

$$S = \sqrt{\frac{\Sigma(X - \bar{X})^2}{n - 1}}$$

Where X= sample particle count, or percentage of concentration, $\bar{X}$= average concentration, and n= number of observations. He also suggests the use of a control chart on the average number of particles per sample. The use of control charts (based on the range and/or on the level of X required) for mixing studies is also mentioned by Maurer (21).

Weidenbaum (10), in discussing the state of mixedness of a batch of solids, feels that "a good general measure of the extent of mixedness is the standard deviation among spot samples removed from the batch. The sample mean should also be reported". Using both the standard deviation and the sample mean, as the coefficient of variation, Creamer (22), in working with fertilizer, uses the following measure:

$$C_V = \frac{100S}{M} = \frac{100\sqrt{\frac{\Sigma(Y - M)^2}{n - 1}}}{M}$$

Where S= standard deviation, Y= individual sample value, M= mean, and n= number of samples. He uses the coefficient of variation because:

> This index adjusts the standard deviation of the averages of several series of observations to a uniform basis for comparison. According to the distribution of the normal curve, 68% of the analyses of individual mixture should exhibit variations of $\leq \pm 1C_V$, 95% $\leq \pm$ $2C_V$, and 99% $\leq \pm$ $3C_V$ from the average.

Working with a calcite powder system in which an organic pigment was used as a tracer, Rumpf and Mueller (2) measured mixing by the use of the coefficient of variation. They also added confidence limits on the coefficient of variation to determine when mixing was random. At each time period 8 spot samples were taken from within the mixer. In commenting on the coefficient of variation Patterson (23) says that "For example a deviation of 2 from a mean of 10 is exactly equivalent, as regards variation, to one of 8 from a mean of 40. For comparative purposes this term . . . expresses the standard deviation as a percentage of the mean . . . . "

In reference to the size of the coefficient of variation, that is, the degree of mixing desired, Pfost (24) gives several criteria which should be considered.

> 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.

He defines the coefficient of variation as $V = \frac{S}{m} \times 100$ Where V= coefficient of variation in percent, S= standard deviation of the assay value, m= mean of the assay values. Several graphs are presented showing the relationship of the size of the coefficient of variation on the probability of exceeding a given tolerance, meeting the minimum guarantee of a component when the degree of mixing and excess component varies, finding a larger excess of a component in a sample when mixing is imperfect, and equalling or exceeding a fraction of the mean as related to the number of particles in a sample. In summary he states that

> . . . the total coefficient of variation should not exceed 20% to avoid possible toxic effects. 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%.

In most of the literature on premixing it is recommended that microingredients be premixed in order to get a random distribution of additives (25), (26), (27). This seems like a logical step: but no data was found that presented experimental results showing a significant difference in the distribution of additives due to premixing, although it seems logical that the size of the premix would make a difference in the time needed to reach a random distribution.

Mahoney and Benson (27) believe that

> . . . direct addition of the feed supplement [micro-ingredients] should be avoided: instead, a premix should be prepared consisting of perhaps one part of the feed supplement and ten to fifty parts of a suitable feed ingredient [carrier]. This premix is then incorporated along with the major components to produce the finished feed.

The 1963 Merck Technical Service Bulletin (28) states that "In most mills equipped with vertical mixers, premixing is particularly necessary if uniform feed blends are to be obtained with reasonable blending intervals." The average time required to blend a premix in a vertical mixer is stated to be 30 minutes. The time required for mixing after the addition of the premixed additives "may vary from 8 to 20 or 30 minutes." A poultry science department's (29) published recommendation for mixing follows:

> When premixing in a vertical mixer the recommended mixing time is one hour with a carrier of ground corn to which is added fat or oil before the supplements [vitamins and drugs] are added. In feed mixing, premix supplements to a volume of at least 10 lbs. per ton. The minimum mixing time for a vertical mixer is 15 minutes.

Rathwell (30) in commenting on single and twin screw vertical mixers says that

> One source of difficulty in the vertical mixer is uneven dispersion of the material as it leaves the top of the mixing tube. A common construction combines the normal velocity imparted by the centrifugal action of the vertical screw itself with the action of a flinger bar attached to the screw shaft.

This " . . . may result in stratification or segregation. The material on its downward path toward the cervix of the cone will tend to be classified . . . "

In tests for mixing efficiency a variety of tracers have been used. Schipke (31) recommends "Spherical pellets having a density of 50 lbs/cu. ft. or wooden blocks as tracers. Creger (4) investigated the dispersion of sodium chloride and nitrophenid in ground yellow corn, soybean oil meal, and a complete poultry ration, and decided that

> Sodium chloride can be used with some confidence as a tracer in mixed feeds. When granular materials are being mixed with relatively long particles, the effects of over mixing may be as deleterious as under mixing. Sodium chloride tends to segregate when mixed with a mass of material that has many particles of relatively large size present, such as soybean oil meal or coarsely ground corn.

With a twin shell blender and a uniform carrier material of solvent extracted soybean oil meal and dyed particles of various materials (beet pulp, solvent extracted soybean oil meal, di-calcium phosphate and ground rock phosphate) Kabance (32) reported the following:

> When tracer was the same material and of the same size or larger than the carrier, no clear-cut evidence was obtained to indicate non-random mixing. Blending tracer of the same material and density but smaller in size than carrier, a marked tendency to segregate in the first few samples [taken from the mixer discharge] was observed. A similar tendency was likewise observed in blending tracers of different densities but material of intermediate size between carrier and tracer was effective in improving the physical factors affecting segregation. Particle size is likely the most important physical factor influencing segregation; however, density may become a limiting factor when there are extreme differences in material blended.

One per cent vegetable oil added to the soybean oil meal carrier also helped to reduce segregation.

Shea and Chandler (33) in working with various feedstuffs using fluorescent and radioisotope tracers reported that particle size differences in feed ingredients should be minimized: bulk ingredients seemed to govern the time of mixing.

The use of drug assays to determine mixing time is recommended by Wornick and others. The analyses for iron and maganese are, according to Pierce (34),

> . . . specific, reliable and relatively easy to use . . . and can be obtained with particle sizes similar to many other additives. These methods are useable as tracers for drugs or additives . . .

Luhman (35) with experimental data showed that the potentiometric determination of soluble chlorides "make it extremely adaptable to the routine analysis of soluble chlorine in feeds."

MATERIALS AND METHODS

Mixer

The mixer used in this research was a 2 ton, top loading, twin screw vertical mixer manufactured by the Prater Pulverizer Company, (Figure 1). It was powered by two 7½ horse power motors with an average speed of 292 rpm. The working capacity was approximately 151 cubic feet. Four probe holes for taking samples from within the mixer were located as shown in (Figure 1).

Sampling

Samples were taken from the mixer by the use of a grain probe with slots three inches long, one inch wide and two inches apart. The first three slots were divided into compartments and used to collect spot samples. Samples were taken at various times, but usually at one or all of the following: 2 minutes, 4 minutes, 6 minutes, 8 minutes, 16 minutes, and 32 minutes. Each time samples were taken from the mixer a total of twelve spot samples were collected; that is, four probe positions with three spot samples per probe. Ten spot samples were also taken at approximately uniform time intervals during the discharge and in some cases samples were taken from a bin or bag (see Figure 2). The spot samples, each approximately 70-80 grams in weight were placed in glass bottles and held for analysis. Before actual analysis, the spot samples were split by the use of a sample splitter to the approximate size needed.

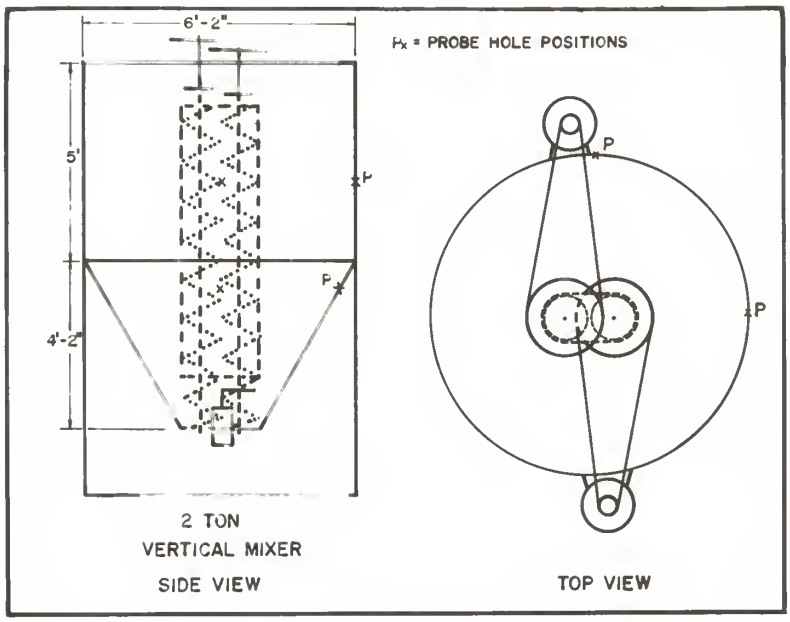

FIGURE 1 SCHEMATIC VIEW OF TEST MIXER

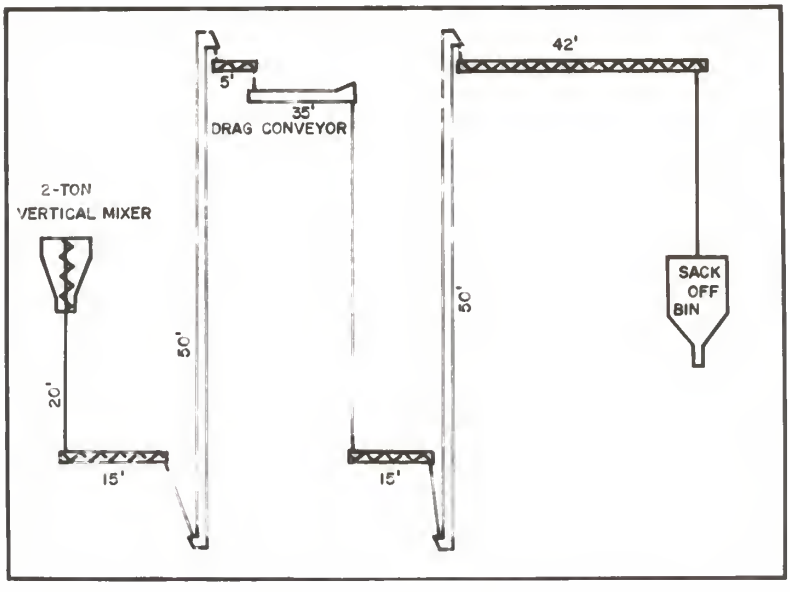

FIGURE 2 FLOW FROM TWO-TON VERTICAL MIXER

Ingredients

Several types of ingredients and in some cases complete rations were used in the experiments. Ingredients used were soybean oil meal, ground milo, ground corn, steam rolled and ground oats, steam rolled barley, a pelleted mineral and protein supplement, salt, limestone, dicalcium phosphate and urea. The complete rations used were a chick grower, a turkey grower, a turkey finisher and a cattle ration.

Procedure

The procedure for mixing was as follows: the bulk ingredients were loaded into the mixer and the tracer, or premix, if used was dumped in all cases on top, through the hatch. After all ingredients were loaded the mixer was started and time was recorded. The mixer was stopped and sampled at the various times as indicated. Upon completion of mixing, the discharge samples were taken from the discharge stream: other samples were taken at other points in the mill as has been indicated.

Methods of Analysis

The following methods of analysis were used:

(a) Ashing method. The A. O. A. C. (1961) method of ashing was followed: the sample size required was 5 grams. Results were expressed as per cent recovery of ash.

(b) Sedimentation method. This test was used to separate the minerals (salt, limestone, dicalcium phosphate) from the rest of the feed mixture. A 1 liter separatory funnel (45 cm. in length) was filled to three fourths capacity with carbon tetrachloride (specific gravity, 1.595) or tetrachloroethylene (specific gravity, 1.623). Thirty grams of feed sample were added and

the funnel was then shaken to release the minerals from the feed. The funnel was left at rest and the mineral ingredients settled to the bottom. At the end of 6 minutes the mineral sediment was flushed through a stopcock into a beaker. The liquid was decanted, the sediment dryed and contents weighed. Results were expressed in per cent of recovery. Data for the sedimentation method recovery variance is given in Table 1. When salt is added to a sample more than 100% sediment is recovered due to foreign material in the feed grain. If salt and limestone are to be recovered the per cent recovery is less than 100%. This is due to the small particle size of the limestone, part of which does not separate from the feed or does not settle out. The coefficient of variation for the method using both salt and limestone is 1.42%.

Table 1. Recovery of salt and limestone by sedimentation assay from individually prepared samples of soybean oil meal, salt and limestone. Total weight, 30 grams, average foreign material was 0.17%.

1% Salt Added			1% Salt and 1% Limestone Added		
Sample Number	% Sediment Found	% Recovery	Sample Number	% Sediment Found	% Recovery
1	1.14	114.	11	1.70	85.00
2	1.10	110.	12	1.79	89.50
3	1.13	113.	13	1.72	86.00
4	1.11	111.	14	1.73	86.50
5	1.15	115.	15	1.75	87.50
6	1.11	111.	16	1.76	88.00
7	1.15	115.	17	1.76	88.00
8	1.14	114.	18	1.75	87.50
9	1.15	115.	19	1.75	87.50
10	1.18	118.	20	1.73	86.50
$\bar{X}$= 1.136, C. V. = 2.10, S= .0238			$\bar{X}$= 1.74, C. V.= 1.42, S= .025		

(c). The potentiometric method. The potentiometric method for the determination of soluble chlorine in feeds, as given by Luhman (35) was used. Results were expressed as per cent salt, sodium chloride; approximate sample size was 5.33 grams. Recovery data for the potentiometric method is tabulated in Table 2. Table 3 shows the recovery in a mixture of barley and pelleted supplement ground for assay: total sample size was 4 pounds and subsample size was 5.33 grams.

Table 2. Recovery data from prepared samples of ground milo and salt. Chloride analysis.

Sample	Sodium Chloride Present,%	Sodium Chloride Added,%	Sodium Chloride Found,%	Recovery,%
Ground Milo	.07	.25	.33	103.12
"	.07	.50	.49	85.96
"	.07	.75	.81	98.78
"	.07	1.00	1.11	103.74
"	.07	1.25	1.22	92.42

Table 3. Assay variation of potentimetric chloride method within a sample of ground rolled barley and pelleted supplement.

Subsample	% Salt	Subsample	% Salt
1	.361	6	.383
2	.402	7	.375
3	.350	8	.405
4	.375	9	.390
5	.382	10	.406

$s = .0182$, C. V. = 4.76

$\bar{X} = .382$

(d) Kjeldahl method. The official method of the A. O. A. C. (1960), as the improved method used for nitrate free samples. The sample size needed for analysis was .9 of a gram.

(e) Amprolium. The amprolium method of Szalkowski and Schulz (36) was used. Sample size was 10 and 15 grams.

(f) Sieve analysis. The method of determining modulus of uniformity and modulus of fineness of ground feed as recommended by the American Society of Agricultural Engineers (37).

The modulus of fineness is a number system indicating the fineness of a material. It is calculated from the amount of material retained on wire sieves. The smaller the modulus of fineness the finer the material.

The modulus of uniformity indicates as a ratio the proportionate amounts of coarse, medium and fine material of a sieved sample. Three figures, the sum of which must equal 10, expresses these proportions.

Statistics

The coefficient of variation was used as a measure of mixing:

$$C.V. = \frac{\sqrt{\frac{\Sigma (X_i - \bar{X})^2}{n - 1}}}{\bar{X}} \times 100$$

Where C.V. = coefficient of variation in per cent
X_i = value of the ith sample
$\bar{X}$ = average of the sampled values
n = total number of samples

To determine the effects of sample variation within the mixer, an analysis was run on several types of mixes. The major variables were time, probe location, and samples within the probe location.

The coefficient of correlation, r, was used to measure the closeness of the linear relationship, between different methods of analyses, r will vary from 0 to 1, where r = 1 denotes perfect linearity.

$$r = \frac{\Sigma x_1 x_2}{\sqrt{(\Sigma x_1^2)(\Sigma x_2^2)}}$$

Where x_1^2 = sum of squares for the variable.

x_2^2 = sum of squares for the variable.

$x_1 x_2$ = the sum of the product of the deviation of each term from its mean.

The t test was used to test for significant differences between treatment means. The tested hypothesis is: $\bar{X}_1 = \bar{X}_2$. The formulae are as follows: (a) for equal n;

$$t = (\bar{X}_1 - \bar{X}_2)\sqrt{\frac{n(n-1)}{\Sigma x^2}}$$

Degrees of freedom are 2(n - 1). (b) for unequal n;

$$t = (\bar{X}_1 - \bar{X}_2)\sqrt{\frac{n_1 n_2 (n_1 + n_2 - 2)}{(n_1 + n_2)\ \Sigma x^2}}$$

Where: $\bar{X}$ = mean X.

n = size of each group.

x^2 = pooled sum of squares.

Degrees of freedom are $(n_1 + n_2 - 2)$.

To determine whether there were any significant differences between certain treatment variances an F test was used. Where the calculated F was:

$$F = \frac{s_x^2}{s_y^2} \qquad \text{if } s_x^2 > s_y^2$$

$$F = \frac{s_y^2}{s_x^2} \qquad \text{if } s_y^2 > s_x^2$$

And:

s_x^2 = the variance of x.

s_y^2 = the variance of y.

RESULTS

Correlation of various assay methods

The coefficient of variation, as a measure of mixing, was used to correlate the various methods of analysis. Correlations were run at each time, at the discharge period and over all periods, in order that any differences due to time and/or mixing would be readily apparent. Figure 3 shows that correlation is good in the early stages of mixing and rather poor after mixing is complete at 8 minutes. After mixing is complete both analyses usually have low values of coefficients of variation. With such little spread in the data, due to the fact that mixing is complete, the correlation at these times is poor and they also tend to reduce the correlation over all sampling periods. The correlation of the sedimentation analysis with amprolium, over all sampling periods, was 0.82.

When the 16 minute, 32 minute and the discharge sampling intervals are left out of the correlation calculations, the correlation coefficient, r, is 0.90. The results of the correlation up to and including 8 minutes will be called the adjusted total correlation coefficient. The adjusted total correlation coefficient of the chloride analysis and the amprolium analysis is 0.75, Figure 4. Correlations were also run between ash and amprolium (see Figure 5). Other correlation data is given in Tables 4 and 5 and in Figures 6 and 7.

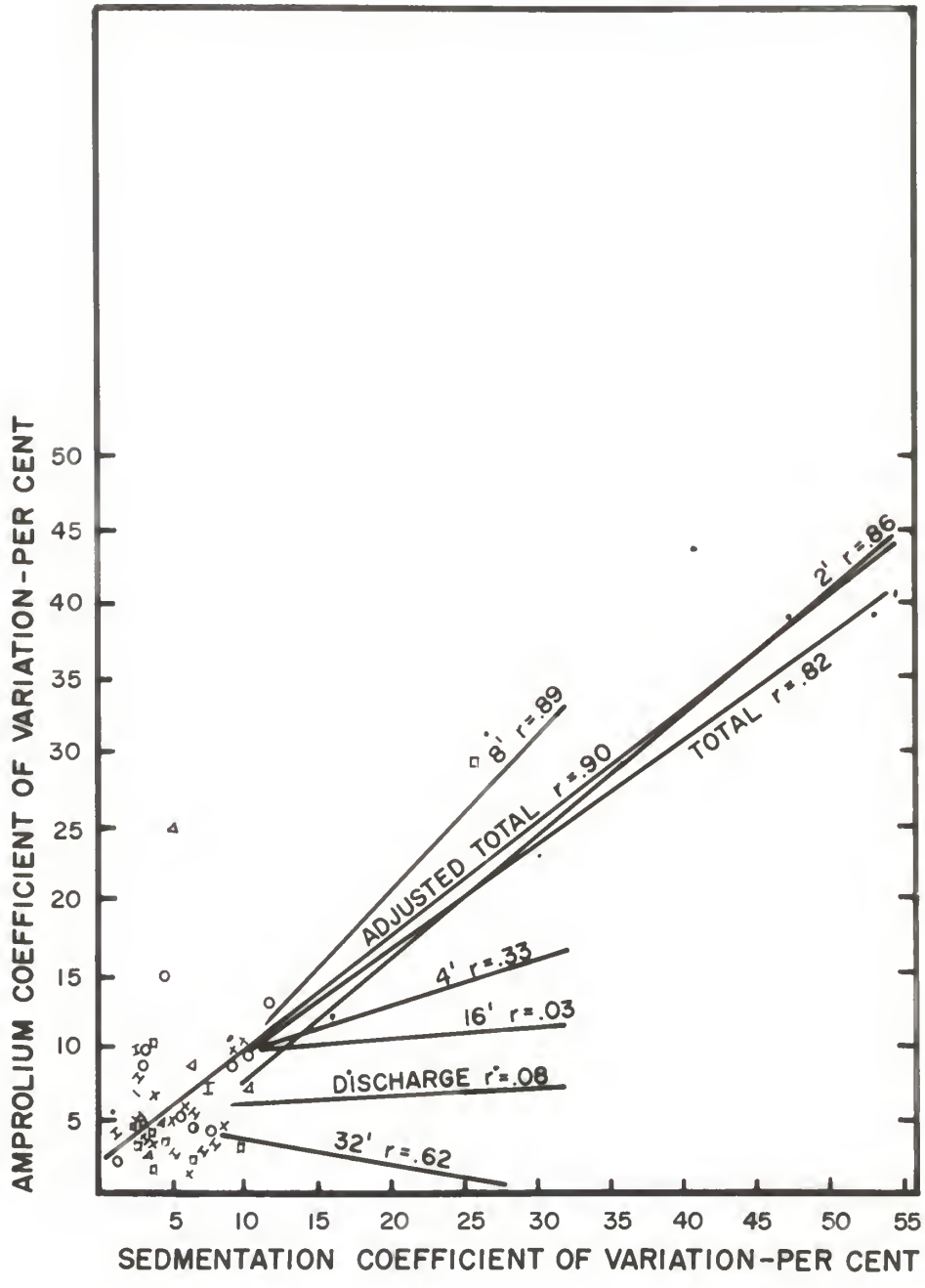

FIGURE 3 RELATIONSHIP OF SEDIMENTATION TO AMPROLIUM ASSAYS AT VARIOUS TIME INTERVALS.

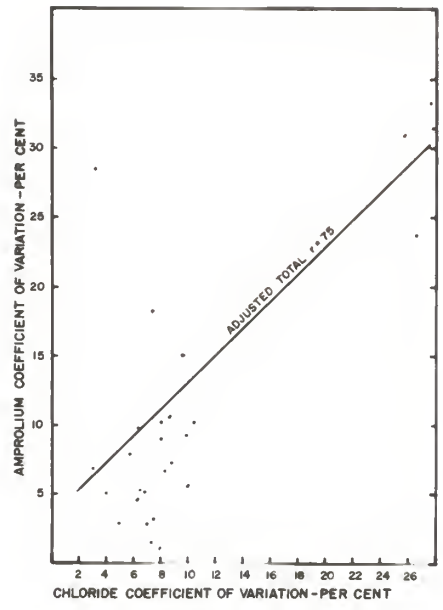

FIGURE 4 RELATIONSHIP OF CHLORIDE TO AMPROLIUM ANALYSIS.

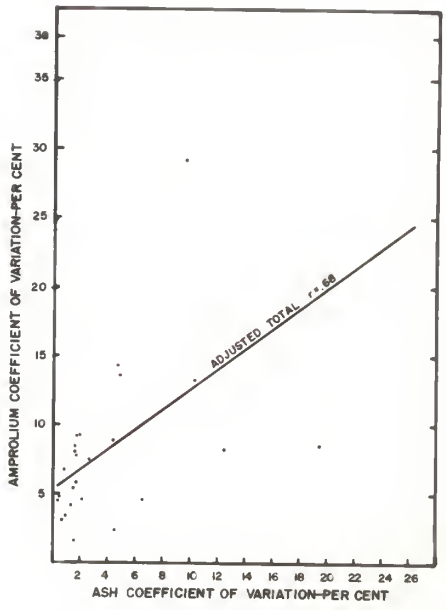

FIGURE 5 RELATIONSHIP OF ASH TO AMPROLIUM ANALYSIS

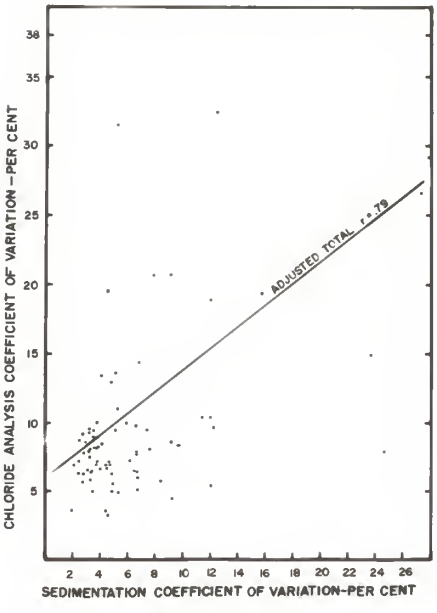

FIGURE 6 RELATIONSHIP OF SEDIMENTATION TO CHLORIDE ANALYSIS.

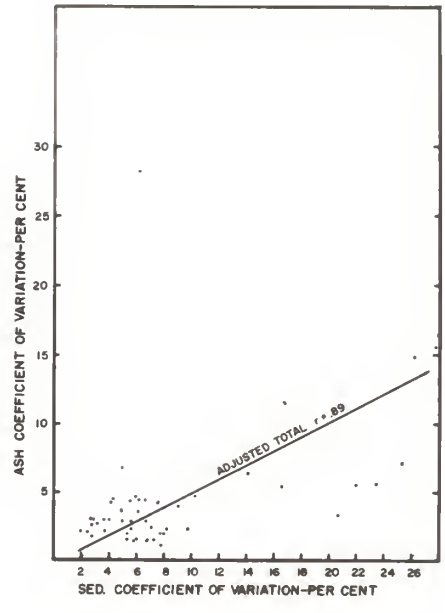

FIGURE 7 RELATIONSHIP OF SEDIMENTATION TO ASH ANALYSIS

The ashing method does not correlate well with amprolium. This is probably due to the effect of small sample size and the large contribution of sample ash made by the grain portion of the sample.

Table 4. Correlation coefficient, r, for amprolium analysis versus sedimentation, chloride and ash analyses.

	Amprolium									
	2	4	8	Adj. Total	Slope	16	32	Disch.	Total	Slope
Sed.	.86	.33	.89	.90	.77	.03	.62	.08	.82	.71
Cl.	.96	.26	.86	.75	.98	.55	.05	.45	.72	1.04
Ash.	.38	.14	.04	.68	.73	--	--	.35	.70	.73

Table 5. Correlation coefficients, r, for sedimentation analysis versus chloride and ash analyses.

	Sedimentation									
	2	4	8	Adj. Total	Slope	16	32	Disch.	Total	Slope
Cl.	.75	.60	.15	.79	.77	.21	.03	.20	.75	.75
Ash	.94	.15	.01	.89	.52	.42	.66	.14	.54	.36

Effects of Mixing Time

Of great concern to the feed manufacturer, and the most important and necessary information about the performance of a feed mixer, is the length of time required to mix a ration which normally could be expected to mix thoroughly. A complete poultry ration was selected for this series of tests. The mixing time needed to reduce the coefficient of variation for a complete poultry ration to 5% was observed to be between 4 and 8 minutes,

at normal mixer speed (292 rpm). To determine the mixing time more accurately, samples were taken at 6 minutes and the mixer was discharged and samples were also taken out of the discharge. The results of the 6 minute mixing time is compared with 8 minute and 32 minute periods in Figure 8. The data shows that 6 minutes was long enough with this mixer and even somewhat conservative. A t test between the means of 4,6, and 8 minutes mixing times failed to show any significant differences due to mixing time.

Table 6. Values of t test for determination of mixing time (.05 level), sedimentation analysis.

Minute		Minute	Cal. t	Table t
8	vs	6	.086ns	2.45
8	vs	4	1.81 ns	2.26
6	vs	4	1.91 ns	2.26

ns = nonsignificant

The following mixtures also met the above criteria and are considered mixed at 6 minutes: (a) ground milo, 1% salt, and 1% limestone and (b) soybean oil meal (ground through a $\frac{1}{2}$ inch screen), 1% salt, and 1% limestone.

Effect of a Differential in Speed of the Screws

The effects of a differential in speed between the mixing screws on mixing were investigated by increasing and decreasing the speed of one of the mixing screws. Four treatments were replicated 3 times each: a control (normal speed), a 36% increase, an 8% decrease and a 13% decrease. The corresponding speeds are

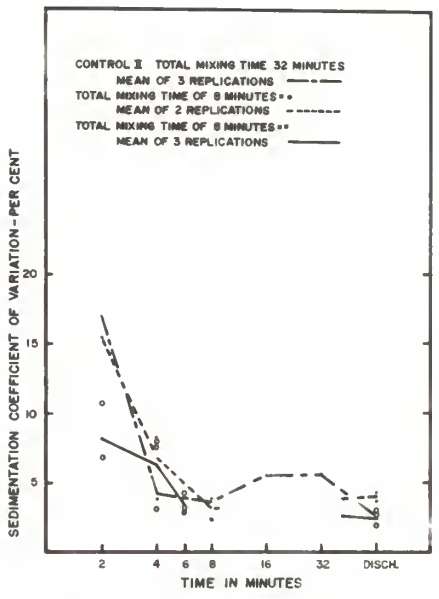

FIGURE 8 DETERMINATION OF MIXING TIME, COMPLETE POULTRY RATION

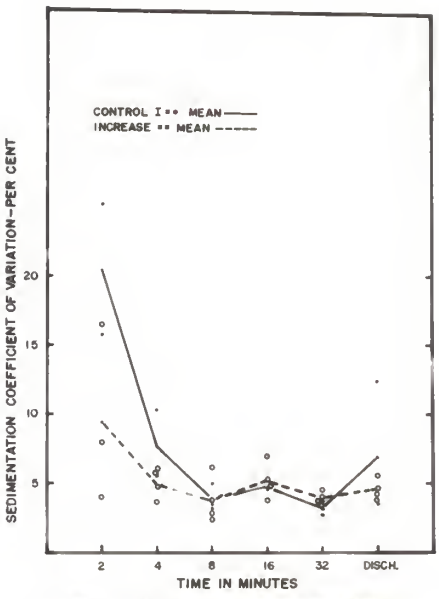

FIGURE 9 THE EFFECTS OF A 36% INCREASE IN SPEED OF ONE MIXING SCREW. CONTENTS: GROUND SORGHUM GRAIN, 1% SALT, 1% LIMESTONE.

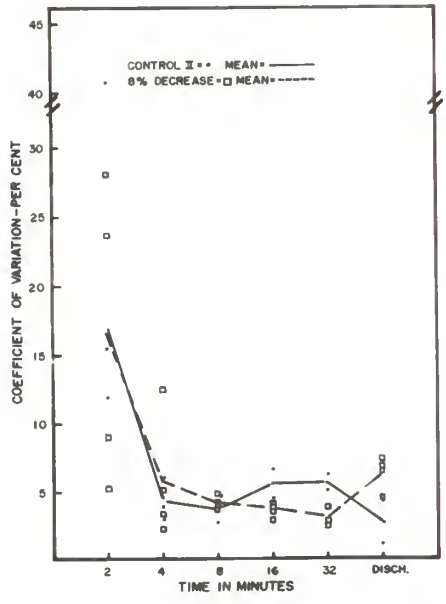

FIGURE 10 THE EFFECTS OF AN 8% DECREASE IN SPEED OF ONE MIXING SCREW. CONTENTS: POULTRY RATION, SEDIMENTATION ANALYSIS.

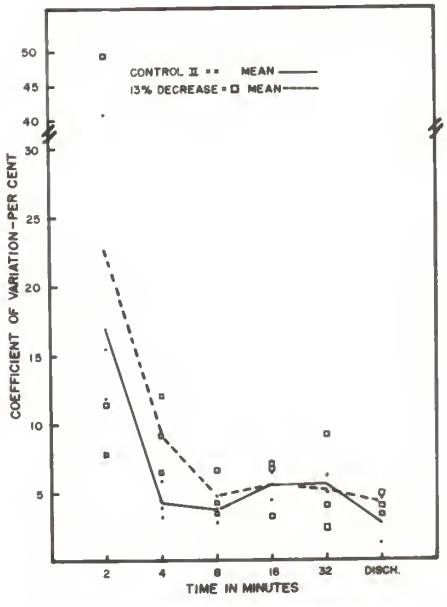

FIGURE 11 THE EFFECTS OF 13% DECREASE IN SPEED OF ONE MIXING SCREW. CONTENTS, POULTRY RATION, SEDIMENTATION ANALYSIS.

294, 400, 270 and 254 rpm. The results are shown in Figures 9, 10 and 11. T tests of the treatment means are evaluated in Table 7.

Table 7. Values of t for change of speed (.05 level). Sedimentation analysis.

	Control I			
	4′ Cal. t	Table t	8′ Cal. t	Table t
36% Inc.	1.88 ns	2.78	.112 ns	2.78

	Control II			
	4′ Cal t	Table t	8′ Cal. t	Table t
8% Dec.	.52 ns	2.57	.66 ns	2.57
13% Dec.	2.58 ns	2.78	1.08 ns	2.77

ns= nonsignificant

The only comparison made that approaches significance is between the control and the 10% decrease at 4 minutes mixing time, which indicates that a slower speed increases mixing time. However, an F test at the .01 level of significance found a highly significant difference between the variances of the 36% increase in speed and the control at the 2 minute mixing period. The plot of the 36% increase versus the control shows that the optimum speed for this mixer and this ration has not been obtained: that is, faster mixing would probably occur if the speed of both mixing screws was increased.

Effect of Premix Size

It was felt that the size of a premix would substantially affect the time required for mixing, as a result, three premix sizes were run. A control with 31% premix which corresponds to the conventional supplement, an 11% premix which corresponds to the normal premix size in a complete feed, and one with no premixing in which microingredients were added without any dilution. They will henceforth be noted as Type I, Type II, and Type III respectively. The poultry formula used and a list of the premix ingredients is given in Table 8. Typical sieve analyses of a complete ration are shown in Figure 12; Table 9 shows size analysis data. Particle size analysis of the mineral ingredients compared to the entire ration is shown in Figure 13.

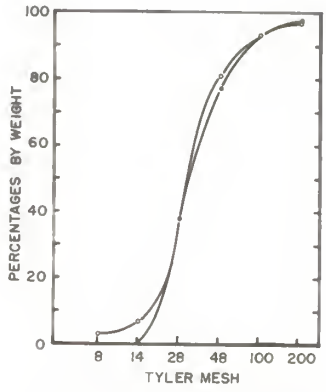

FIGURE 12 CUMULATIVE SCREEN ANALYSIS OF POULTRY RATIONS

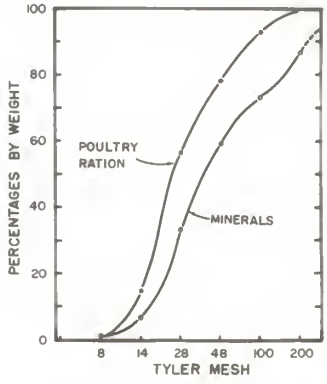

FIGURE 13 CUMULATIVE SCREEN ANALYSIS OF A POULTRY RATION AND ITS MINERAL CONTENTS.

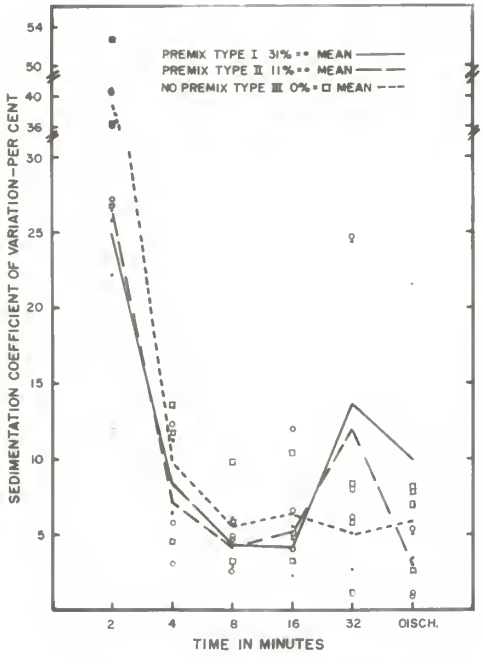

FIGURE 14 THE EFFECT OF PREMIX SIZE ON MIXING

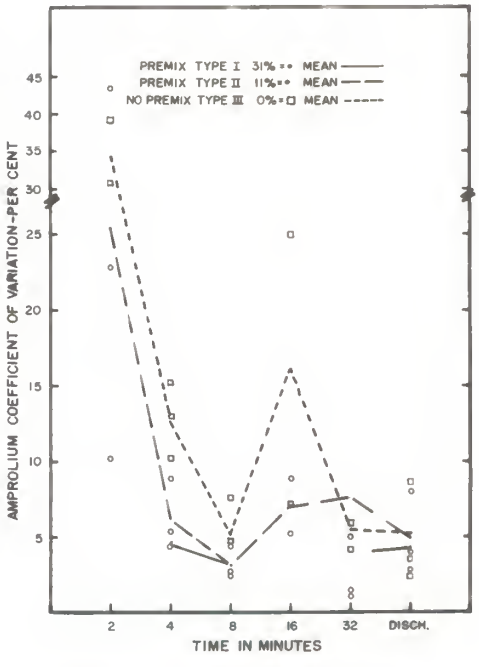

FIGURE 15 THE EFFECT OF PREMIX SIZE ON MIXING

Table 8. Formula for complete poultry ration and ingredients used in premixes.

Poultry Ration
Chick Grower

Ingredient	Amount/ton (lbs)	Ingredients used in Premix		
		I	II	III
Soybean Oil Meal	300	x		
Ground Yellow Corn	500			
Ground Sorghum grain	500			
Ground Oats	200			
Wheat Middlings	200			
Dehydrated Alfalfa Meal	100	x		
Meat and Bone Meal	100	x	x	
Fish Meal	50	x	x	
Ground Limestone	20	x	x	
Dicalcium Phosphate	20	x	x	
Salt	10	x	x	
Trace Minerals	1	x	x	
Vitamin & Drug Premix	20	x	x	
Total weight of ingredients	2021 lbs.	626 lbs.	226 lbs.	0
Percent, by weight, of premix		31%	11%	0%

All ingredients placed in mixer without premixing

Table 9. Particle size analysis of ingredients used.

Ingredient	Fineness Modulus	Modulus of Uniformity
Steam Rolled Oats	4.82	9:10
Steam Rolled Barley	5.06	10:00
Pellets	5.93	10:0:0
Crumbles (1)	4.30	5:5:0
" (2)	4.15	4:6:0
" (3)	4.08	3:7:0
" (4)	3.80	1:9:0
Amprolium	1.96	0:2:8
Limestone	.68	0:0:10
Salt	1.89	0:0:10
Urea	1.65	0:1:9
Soybean Oil Meal	2.83	0:7:3
Ground Soybean Oil Meal	2.07	0:4:6

The plot of the means of three replications of the sedimentation analysis (Figure 14) is about what would logically be expected. In an F test at the .05 level of the 2 minute time period treatment variances, significant differences were found between: Type III and Type I, and at the .10 level between Type III and Type II. No significant differences were found between the variances of Type II and Type III at the .05 or .10 levels of significance.

Table 10. F Tests of premix size effect at 2 minutes of mixing. Sedimentation analysis. Variances of all samples, means assumed equal.

Treatments	P	Table F	Calc. F
Type III vs. Type I	5%	1.79	1.88 *
	10%	1.59	" *
Type III vs. Type II	5%	1.79	1.60 ns
	10%	1.58	" *
Type I vs. Type II	5%	1.82	1.17 ns
	10%	1.61	" ns

ns = nonsignificant * = significant

Further statistical analysis by t test showed no significant differences between the three treatment means at 4 and 8 minutes of mixing (Table 11). This would indicate that mixing is so rapid as to obliterate any differences before 4 minutes of mixing and that the size of the premix makes no practical difference on the time of mixing

Table 11. Values of t for premix sizes, (.05 level). Sedimentation analysis.

	Type I (Control)					
	2'-Cal.t	Table t	4'-Cal.t	Table t	8'- Cal.t	Table t
Type II	.568ns	2.78	1.29 ns	2.78	.24 ns	2.78
Type III	1.74 ns	"	.498ns	2.78	.83 ns	"

ns = nonsignificant

Amprolium analyses were made on part of the tests (Figure 15), and these tests tend to substantiate the sedimentation analyses. Only the t test at 4 minutes between the means of Type II and Type III (Table 12), shows a significant difference between the treatment means in favor of the larger premix. The 25% -activity amprolium was added at a level of 1 pound per ton of the total ingredients.

Table 12. Values of t for Type II Premix vs. Type III. Amprolium analysis . (.05 level)

2′ Cal. t	Table t	4′ Cal. t	Table t	8′ Cal.T	Table t
.74 ns	3.18	3.28*	2.78	1.34ns	2.78

ns = nonsignificant
* = significant

Problem Ingredients
Soybean Oil Meal

Results of exploratory tests showed that minerals such as salt and limestone did not mix well with such ingredients as soybean oil meal and rolled grains. A mixture of soybean oil meal, 1% salt, and 1% limestone, was investigated because of these segregating tendencies. It was hoped that grinding part or all of the soybean oil meal would reduce or eliminate this tendency. Four treatments were run with replications: an unground control, and with 10%, 20%, and 100% of the soybean oil meal ground through a $\frac{1}{4}$ inch hammer mill screen. Sieve size analyses were run as shown in Figures 16 and 17, and Table 9 shows particle size data. The results of grinding are shown in Figures 18 and 19. To determine if there were any significant differences between the treatment means, t tests were used to evaluate them (Table 13). The mean of the 100% ground treatment was significantly different from the control at 8 minutes and the graph indicates better and faster mixing. There were no significant differences between the control and the material with

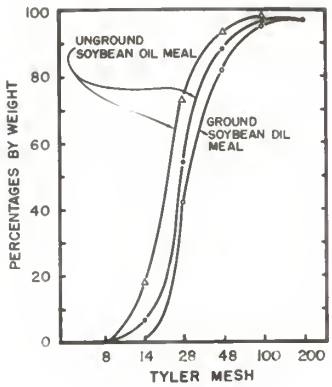

FIGURE 16 CUMULATIVE LOGARITHMIC SCREEN ANALYSIS ON SAMPLES OF UNGROUND AND GROUND SOYBEAN OIL MEAL.

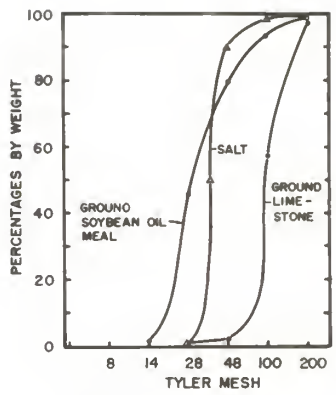

FIGURE 17 CUMULATIVE SCREEN ANALYSIS OF GROUND LIMESTONE, GROUND SOYBEAN OIL MEAL, AND SALT.

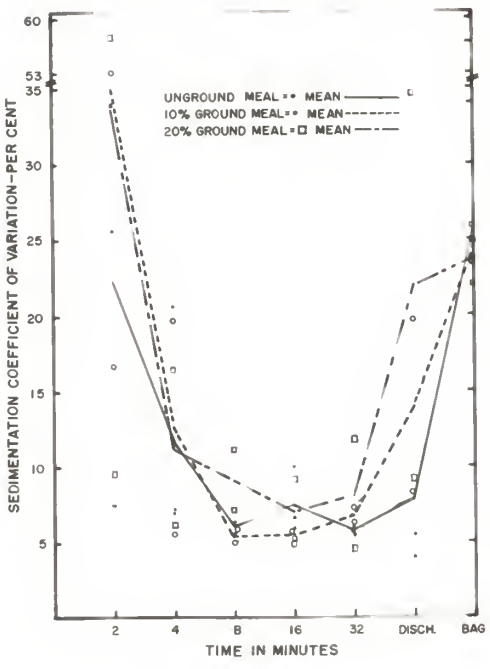

FIGURE 18 THE EFFECT ON MIXING AND SEGREGATION OF GRINDING SOYBEAN OIL MEAL THROUGH A 1/4" HAMMER MILL SCREEN, IN A MIXTURE WITH SALT AND LIMESTONE.

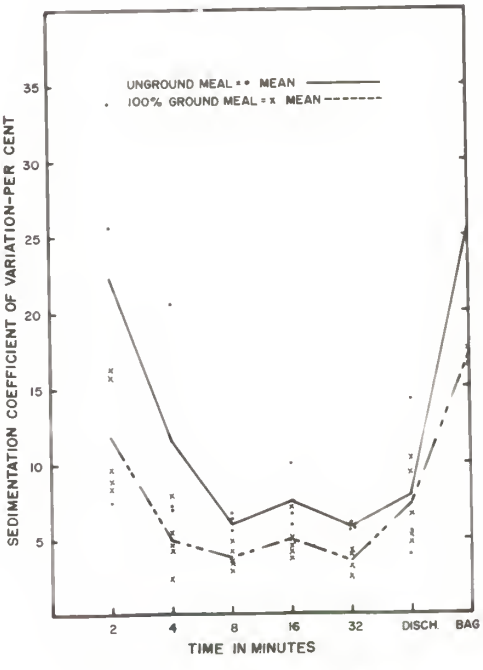

FIGURE 19 THE EFFECT ON MIXING AND SEGREGATION BY GRINDING SOYBEAN OIL MEAL THROUGH A 1/4" HAMMER MILL SCREEN IN A MIXTURE WITH SALT AND LIMESTONE.

10% or 20% reground. From Figure 19, it is indicated that the 100% ground material gave less segregation after mixing due to handling and bagging operations.

Table 13. Values of t for soybean oil meal, salt & limestone system (.05 level). Sedimentation analysis.

	Soy 0% Ground				
	df.	4- Cal. t	Table t	8- Cal. t	Table t
10% gr.		.124ns	3.18	1.58ns	3.18
20% gr.		.063ns	3.18	2.02ns	"
100% gr.		1.88 ns	2.45	4.77 **	2.45

NS = nonsignificant
** = highly significant

Rolled Grain

To determine the problems encountered in mixing rolled grain with other products of smaller particle size, a 3,000 pound mixture of steam rolled oats, urea, salt and limestone was tested. The amounts of the ingredients are given in Table 14.

Table 14. Formula of oats mixture

Rolled Oats	93%
Urea	5%
Salt	1%
Limestone	1%
	100%

Figure 21 shows the results of sedimentation and Kjeldahl analyses. Particle size analyses are shown in Figure 20. It is clear from Figure 21 that the segregating tendencies of the minerals and the urea from the grain was very great, with the minerals having approximately double or more the coefficient

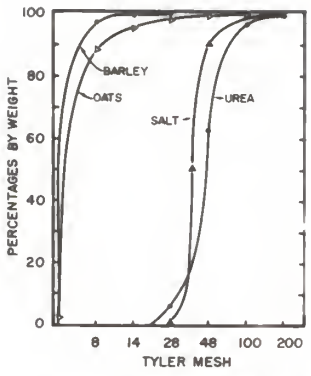

FIGURE 20 CUMMULATIVE SCREEN ANALYSIS OF UREA, BARLEY, OATS, AND SALT.

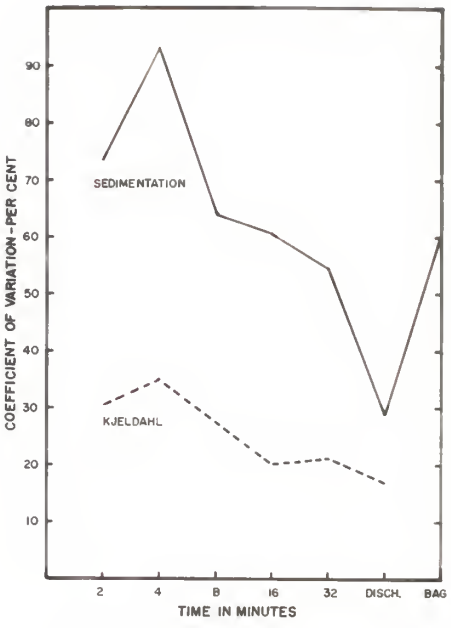

FIGURE 21 EFFECTS OF MIXING ROLLED OATS, UREA, SALT AND LIMESTONE.

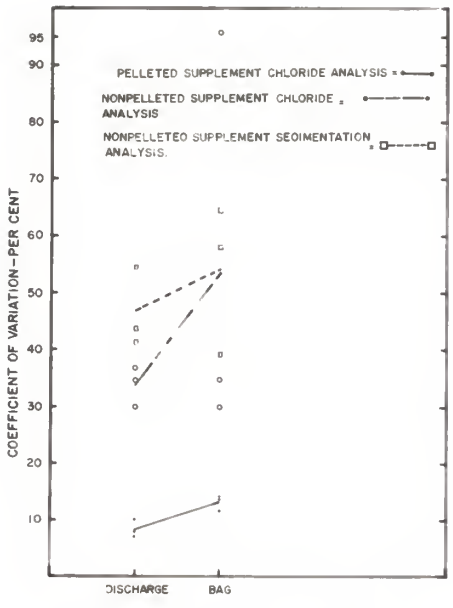

FIGURE 22 EFFECT ON MIXING AND SEGREGATION OF PELLEING A PROTEIN-MINERAL SUPPLEMENT TO BE MIXED WITH A STEAM ROLLED GRAIN CONTENTS: STEAM ROLLED BARLEY, SOYBEAN OIL MEAL, SALT, AND LIMESTONE.

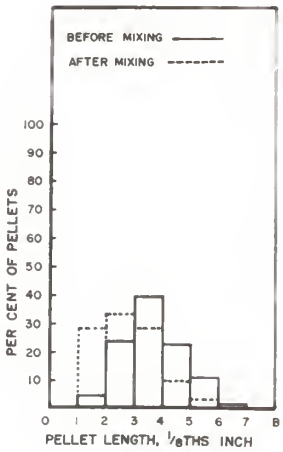

FIGURE 23 EFFECT OF MIXING PELLETS ON PELLET LENGTH.

of variation, of about 17%.

A similar problem to that of rolled oats is one of a protein-mineral supplement with steam rolled barley. A mixture of steam rolled barley and a supplement, which was added at a level of 5% to the grain, was tested. The formula is shown in Table 15.

Table 15

Protein Mineral Supplement	
Soybean Oil Meal	71%
Salt	15%
Limestone	14%
	100%

Formula of barley & supplement mixture used for testing rolled barley ration	
Barley	95%
Protein mineral supplement	5%
	100%

Exploratory work was done with a supplement added to rolled grain in a transparent twin shell mixer. The supplement was added in the following forms: test 1, loose mash form; tests 2, 3, 4, and 5 four sizes of crumbles, and test 6, as 3/16 inch pellets. Modulus of fineness and modulus of uniformity are given in Table 9. The pelleted form was observed to mix better than the others, and was chosen for use in the vertical mixer tests.

The control for these tests was the supplement added in loose form. Three replications of each treatment were made with each test consisting of 2,000 pounds of the mixture. The results of sedimentation and chloride analyses are given in Figure 12. The t tests, as shown in Table 16, indicate significant differences at the discharge, but not in the bag samples. The reason for the latter is the error of the t test which is due to the large inequality of the variances. In the case of the bag off samples an F test was used to evaluate the treatment variances. The F test at the .01 level found a very highly significant difference between the pelleted and non-pelleted supplements. It is therefore evident that the use of a pelleted supplement will give an acceptable mix which does not segregate excessively during subsequent handling.

Table 16. t Table for barley & supplement system, chloride analysis, .05 level

	Pelleted Supplement			
	Discharge at 8-		Bag off	
	Cal. t	Table t	Cal. t	Table t
Loose Supplement	13.85***	2.78	1.88 ns	2.78

***=very highly significant

The quality of pellets used in this type of mixture could be expected to effect the amount of fines produced and thus the distribution of the supplement in the mixture. In order to maintain pellet quality during these tests the following criteria

was followed and all supplements to be used in the tests were pelleted at one time. The conditioning temperature of the supplement during pelleting was 80 to 85 degrees centigrade. The pellet durability index (36) was 9.62. Samples of pellets were taken before and after mixing and measured to determine the distribution of their length. This data is shown in Figure 23. The size of the sample to be taken at the discharge and the sack off was determined by counting 1,000 pellets, weighing them and calculating the expected number per pound of ration, which was approximately 75.67 pellets. Because the sampling variance due to the Poisson distribution of such a small number of pellets is quite large, a 4 pound sample was taken in which the theoretical coefficient of variation in per cent due to the Poisson distribution is:

$$C.V. = \frac{100}{\sqrt{m}} = \frac{100}{\sqrt{302.8}} = 5.75\%$$

Where m is equal to the expected mean number of particles,

Analysis of Variance

Typical results of the analyses of variance on several mixes are shown in Table 17. In general, the analysis of variance shows little or no significant differences in regard to stratification or segregation due to the mixer, but rather indicate that problems of mixing are due to properties inherent in ingredients, such as soybean oil meal.

Table 17. Analysis of variance for effect of location, sample and time on mixing at the .05 level of significance. Sedimentation analysis.

Ground Sorghum Grain

SOURCE OF VARIATION	DF	F	
LOCATION L	5	6.37	**
SAMPLES S	2	.99	ns
TIME T	4	.02	ns
L X S	6	.80	ns
L X T	12	4.26	**
S X T	8	.69	ns
ERROR	24		
TOTAL	59		

Ground Corn

SOURCE OF VARIATION	DF	F	
LOCATION L	3	2.15	ns
SAMPLES S	2	.18	ns
TIME T	4	2.22	ns
L X S	6	.26	ns
L X T	12	.91	ns
S X T	8	.56	ns
ERROR	24		
TOTAL	59		

Poultry Ration

SOURCE OF VARIATION	DF	F	
LOCATION L	3	11.07	**
SAMPLES S	2	2.64	ns
TIME T	4	3.19	*
L X S	6	.67	ns
L X T	12	9.45	**
S X T	8	.98	ns
ERROR	24		
TOTAL	59		

ns= nonsignificant
* =significant
**=highly significant

Table 17. Analysis of variance for effect of location, sample and time on mixing at the .05 level of significance. Sedimentation analysis (continued)

10% Ground Soybean Oil Meal

SOURCE OF VARIATION	DF	F	
LOCATION L	3	4.42	*
SAMPLES S	2	5.31	*
TIME T	4	1.49	ns
L X S	6	1.36	ns
L X T	12	5.86	*
S X T	8	.41	ns
ERROR	24		
TOTAL	59		

Unground Soybean Oil Meal

SOURCE OF VARIATION	DF	F	
LOCATION L	3	25.76	***
SAMPLES S	2	4.01	*
TIME T	4	11.56	**
L X S	6	1.64	ns
L X T	12	20.67	**
S X T	8	1.02	ns
ERROR	24		
TOTAL	59		

20% Ground Soybean Oil Meal

SOURCE OF VARIATION	DF	F	
LOCATION L	3	3.37	*
SAMPLES S	2	2.33	ns
TIME T	4	.81	ns
L X S	6	1.72	ns
L X T	12	1.33	ns
S X T	8	.39	ns
ERROR	24		
TOTAL	59		

*** = very highly significant

CONCLUSIONS

1. Mixing a ration with the major ingredients similar to a complete poultry ration in the 2 ton, twin screw, vertical mixer tested in this research project was complete in 6 minutes or less, if completeness is defined as a coefficient of variation of 5% or less.

2. The sedimentation test measures the mixing of minerals and also correlates well with amprolium up to the point where mixing is considered complete. It is, therefore, considered as a valid means of testing feed mixers.

3. The potentimetric method for soluble chloride can be used to measure the mixing of salt in feeds and correlates well with amprolium to the point where mixing is considered complete.

4. The ashing method will follow the mixing of the minerals until mixing is complete, but does not correlate as well as the sedimentation and chloride analyses with amprolium.

5. Work with a commercial source of soybean oil meal indicated that grinding 100% through a $\frac{1}{8}$ inch hammer mill screen will solve most of the segregation problems during mixing and handling. The particle size of soybean oil meal as received by many feed manufacturers is not of optimum size, especially for use in simple mixtures where it forms the bulk of the ingredients. The analyses of variance on the different treatments tends to substantiate the above conclusions. It also points out the fact that the bulk ingredients definitely govern the time and thoroughness of mixing, provided that the critical ingredients

remain the same.

6. Pelleting an added supplement will vastly improve its mixing properties with a steam rolled grain such as barley.

7. Two ways of improving mixing of problem ingredients or mixtures are: (a) reduce the particle size of the bulk ingredients or (b) increase the effective particle size of the additives by means such as pelleting.

8. The size of a premix in a 2 ton mixer that would mix in 6 to 8 minutes made little difference in time, or thoroughness of mixing.

9. There were no significant differences in mixing time due to an 8% or a 13% speed differential obtained by reducing the speed of one mixer screw. There was a significant difference due to a 36% speed differential obtained by increasing the speed of one mixing screw. The optimum mixing speed for this mixer and the rations tested probably has not been reached, but a speed increase toward optimum would require more power.

10. The coefficient of variation is a useful statistical tool, sophisticated enough to describe the mixed state of two or more materials, and its calculation and useage is simple enough to be easily learned.

SUGGESTIONS FOR FURTHER WORK

It is recommended that more work be done on sizes of premixes using other drugs, or by replicating amprolium. More quick tests for drugs should be developed, as well as more physical tests that will correlate highly with the drugs over a wider range of speed, expecially increases, with both mixing screws would be profitable.

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MIXING STUDIES OF A VERTICAL MIXER AND SOME PROBLEM INGREDIENTS

by

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The importance of mixing is due in large part to the addition of small amounts of various drugs, vitamins and trace minerals to the feed ration. These additives should be distributed as homogeneously as possible, in accordance with the needs of the animal.

With the necessity to meet certain mixing requirements in the animal feed there is a need in the formula feed industry, for first of all a standard measure of mixing, and criteria that defines that which constitutes good mixing, in terms of the standard measure.

Another requirement would include rapid inexpensive tests that would correlate well with drugs, vitamins, and trace minerals in determining the degree of mixing.

The measure of mixing used in this work was the coefficient of variation. It is felt that this statistical measure is sophisticated enough to be useful and yet simple enough that its calculation and use can easily be learned.

The purpose of this research was to study using the coefficient of variation in a vertical mixer the effects of time, a speed differential of the mixing screws, premix size, and ingredient characteristics on the degree of mixing. Fairly rapid and inexpensive methods of analysis were also correlated with a drug to determine their suitability, as at least partial replacement for expensive and time consumming drug assays.

Of great concern to the feed manufacturer about the performance of a mixer, is the length of time required to mix a ration which normally could be expected to mix well. The mixing time needed to reduce the coefficient of variation for 2 tons of a

complete poultry ration to 5% was approximately 6 minutes, at normal mixer speed. Various other mixtures also met the above criteria.

It was felt that the size of a premix used in a feed would substantially affect the time required for thorough mixing, as a result three premix sizes were tested, 31%, 11%, and 0%. In the early stages of mixing a larger premix would do a better job of distributing the micro-ingredients, but for all practical purposes the size of the premix made no significant difference in the time needed to reach a mixed state.

Work with a commercial source of soybean oil meal indicated that grinding through a 1/4" hammer mill screen will solve most of its segregation problems during mixing and handling.

In mixing mixtures of a protein-mineral supplement and a rolled grain, pelleting the supplement will improve its mixing properties.

There was a significant difference in mixing due to a 36% speed differential obtained by increasing the speed of one mixer screw. The optimum mixing speed for the mixer and the rations tested probably has not been reached, but an increase in speed toward optimum would require more power.