the nitrogen solutions would be beneficial in promoting a more uniform reaction, and would tend to promote faster reactions. The use of baffles may not always be warranted.

Operating variables which should be carefully considered in designing commercial reactors include feed rate, particle size, and free moisture content of the superphosphate. Since ammoniation reactions are hindered by large particle sizes, it is desirable from an ammonia reaction standpoint to minimize granulation during ammoniation. The addition of more than 3.7 pounds of ammonia per unit of P_2O_5 will obviously be difficult if low ammonia losses are to be obtained. Good ammonia absorption might be obtained, however, with some type of countercurrent flow of the fertilizer particles with the nitrogen solution. In this respect, the depth of the distributor below the surface of the phosphate fertilizer would be important. Special consideration of these factors may be necessary at higher feed rates of nitrogen solutions to the fertilizer, the use of nitrogen solutions with higher ammonia vapor pressures, or higher temperatures.

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FERTILIZER TECHNOLOGY

Segregation Studies of Dry Blended Fertilizer

L. J. PIRCON, M. de la PUENTE,[1] and MAHDU SHETH[2]
Central Farmers Fertilizer Co., Chicago, Ill.

A study of the mechanism of segregation in dry blended fertilizers has resulted in the mathematical development of a "friction factor" to predict segregation. The mechanics of determining the friction factor by measurement of the angle of repose of free, conical piles of fertilizer ingredients are discussed; and the use of ingredients with matched friction factors are shown by results of experiments to reduce segregation. For example, the almost complete failure of a blend made from materials of random friction factor to meet chemical and physical tolerances is compared to the marked improvement of a blend made from selected materials. Examples of the variation of friction factor with particle size are given for representative materials.

PHYSICALLY blended fertilizers applied in bulk by truck spreader are an economical and widely used form of plant food. However, after spreading, various components of the mixture do not appear in the same ratio at different points in the field. The raw materials have been physically blended, loaded, transported, and spread; and the deficiencies connected with one or more of these operations will show up in the field. In this paper, the segregating mechanisms are analyzed and the conclusions applied to the improvement of the loading and transporting operations, and assistance in raw material specification determination.

A mixture of materials can be considered absolutely nonsegregated only where a crystalline type of structure exists and an elemental pattern is repeated throughout. This situation is impossible in physical mixtures; hence a complete blend is described as a mixture where, for a reasonable volume of material sampled, the chemical composition is independent of the point sampled. The sample size is arbitrary; therefore, the numbers assigned to measure segregation are arbitrary. From taking the total volume of blend (segregation = 0) to taking a unique particle (segregation = 100), there is a complete range of values.

The particles of a blend segregate because the gravitational, vibratory, and resistive forces acting upon them are of different values. The gravitational and vibratory forces relate to the particle's mass, but the resistive depends also on the particle's shape, composition, and surrounding media. This is illustrated by observing the segregation caused by dropping material onto a conical pile and studying the behavior of a particle sliding down the pile (the particle, if rolling, always reaches equilibrium by sliding at the last part of its movement). The gravitational force, P, acting on a particle situated on an inclined surface, can be resolved into

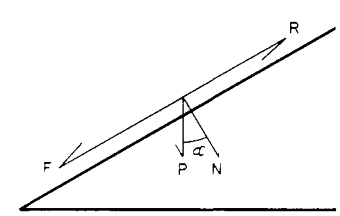

Figure 1. Forces acting on a particle located on an inclined plane

P, gravitational force; α, angle of inclination of the plane; *N*, component of *P* normal to the plane; *F*, component of *P* parallel to the plane; and *R*, resistive force

[1] Present address: Monsanto Chemical Co., Barcelona, Spain.
[2] Present address: International Mineral and Chemicals Corp., Bartow, Fla.

two vectors, F and N, as shown in Figure 1, where $N = P \cos \alpha$ and $F = P \sin \alpha$. If an ideal case is assumed, the particle will descend across the plane due to the action of a force $F = P \sin \alpha$,

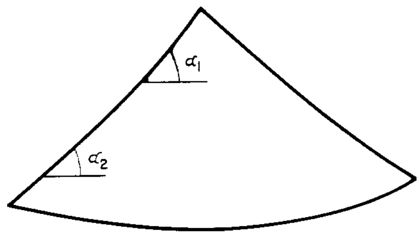

Figure 2. The variation of angle of repose in a conical pile resulting from variation in particle size

α_1 and α_2, angles of repose at different vertical distances from the base

and the corresponding acceleration will be

$$a = \frac{F}{M} = \frac{P \sin \alpha}{M} = \frac{MG \sin \alpha}{M} = G \sin \alpha$$

where a = acceleration, and M = mass. When the plane is vertical, $\sin \alpha = 1$ and, as logical, $a = G$. The resistive force, R, acting against the movement is proportional to the component of the total force, P, perpendicular to the plane

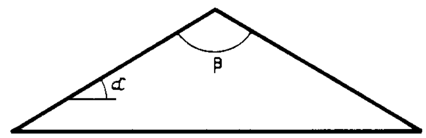

Figure 3. Measurement of angle of repose by measuring vertex of the pile

α, angle of repose; β, vertex angle

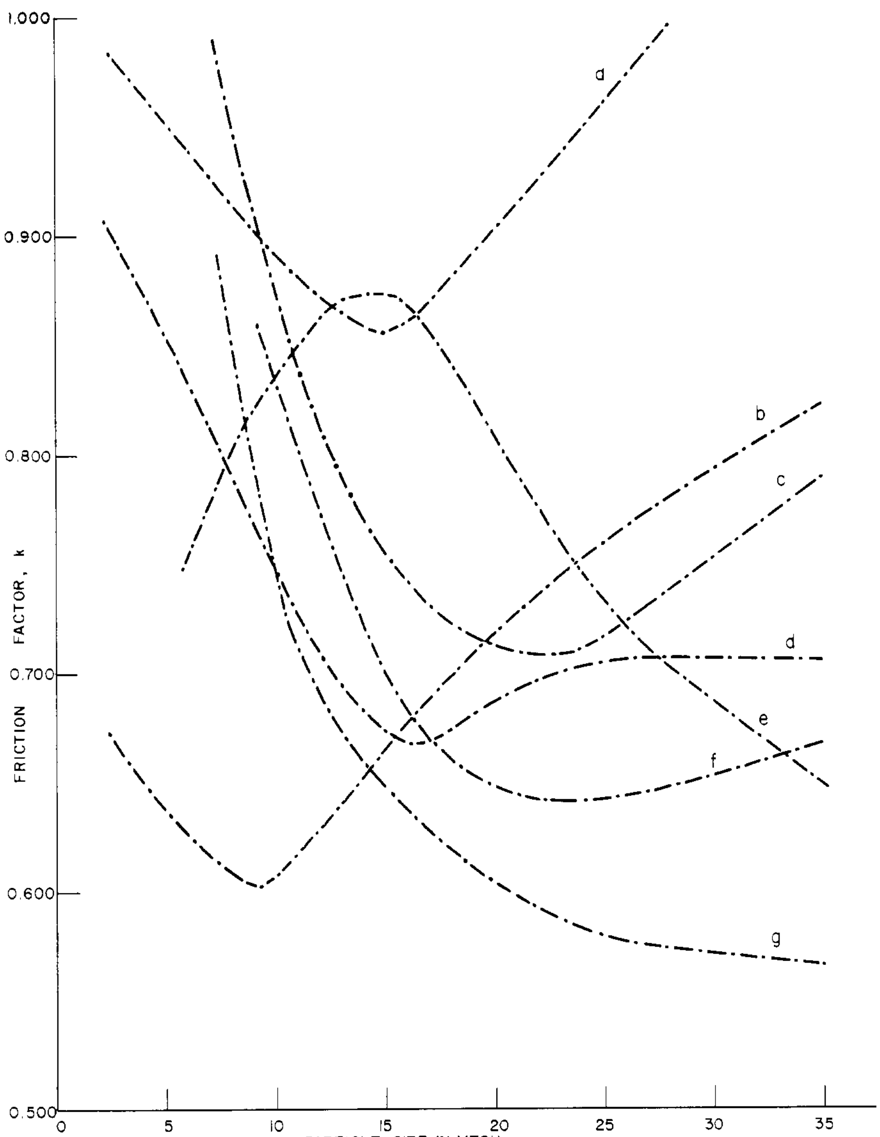

Figure 4. Variation in friction factor, *k*, with variation in particle size for several materials

(*a*) Flaked ammonium nitrate; (*b*) prilled ammonium nitrate; (*c*) compacted triple superphosphate; (*d*) granulated triple superphosphate; (*e*) ammonium sulfate; (*f*) rice-shaped ammonium sulfate; and (*g*) granular potash

N, or $R = kN$. This constant of proportionality, k, is referred to as a friction factor.

The net force acting upon the particle is $(F - R)$, and its acceleration, a, will be

$$a = \frac{F - R}{M} = \frac{MG \sin \alpha - k\ MG \cos \alpha}{M}$$

$$\text{or } a = G(\sin \alpha - k \cos \alpha).$$

There is a certain value of α for which $F = R$ and the particle does not move. At this moment $a = 0$ and $G(\sin \alpha - k \cos \alpha) = 0$. Therefore, $k = \tan \alpha$, a relationship permitting simple measurement of the friction factor.

When a material is piled, the granules move downward until equilibrium is reached. At this point, the surface of the pile achieves its own characteristic inclination, and a careful observation of it shows that the generatrix of the cone is not a straight line (Figure 2). Also, larger particles generally go to the bottom. This is a key observation in attempting to understand the segregation phenomenon. Apparently different sizes of particles have different equilibrium inclinations, or, in other words, different friction factors. The importance of the observation is that in the theoretical explanation given above, the friction factor described is a function of the rugosity of the particle and the rugosity of the plane. Furthermore, this indicates the need for the use of a weighted average k factor to describe the material, because industrially it is not available with all of the particles being the same size but rather comes delivered with the particles spreading over a size range. Nor would it be adequate to select materials to be blended on the basis of equal particle size range distribution, because variables other than particle size affect a specific friction factor value.

As Figure 2 shows, an average k factor could be determined by measuring incremental angles of repose, $\alpha_1, \alpha_2 \ldots \alpha_n$, from the base to the apex of the pile, taking into consideration the mass fraction of the material corresponding to each increment. A practical approach is to physically separate the pile into piles containing particles of nearly equal particle size, determine the angle of repose and mass of each of these smaller piles, and then calculate a weighted average friction factor from individual k's and pile weights as follows:

$$k_{av.} = \frac{w_1 k_1 + w_2 k_2 + \ldots\ldots w_n k_n}{w_t}$$

where w_n = weight of screen fraction n, k_n = k factor of the nth pile, and w_t = total weight of the whole pile containing all size fractions $(w_1 + w_2 + \ldots\ldots\ldots w_n)$. Once the friction factors are determined for different materials, the approach is simplified. Mixed particles will not segregate when they all have the same angle of repose, α.

To what extent particles will segregate can be predicted with the following reasoning. When two particles are rolling down in the same plane, the net forces acting are given by the formulas:

$$F_1 = P_1 (\sin \alpha_1 - k_1 \cos \alpha_1)$$
$$F_2 = P_2 (\sin \alpha_2 - k_2 \cos \alpha_2).$$

As two particles slide down a pile, starting with the same initial speed and initial height

$$a_1 = G (\sin \alpha_1 - k_1 \cos \alpha_1)$$
$$a_2 = G (\sin \alpha_2 - k_2 \cos \alpha_2)$$

After a period of time, t, the relative distance between the two particles will be $\Delta S = S_2 - S_1 = {}^1/_2 t^2(a_2 - a_1) = {}^1/_2 t^2 G (\sin \alpha_2 - \sin \alpha_1 - k_2 \cos \alpha_2 + k_1 \cos \alpha_1)$, where S = distance traveled, t = time. When $k_2 = k_1$, since $k = \tan \alpha$, then $\alpha_1 = \alpha_2$, $\Delta S = 0$, or, in other words, there is no segregation or separation of particles.

A study of transportation segregation is more complicated due to movement of the particles by forces other than gravity. The frequency of vibration and the shape of the container create periodic movements that show up in different forms. One is the packing of the material during initial movement of the truck. The second is a slow motion due to the stationary waves that create one or more maxima of vibration, and then a motion of the particles rising and rolling down past the walls of the truck.

A theory explanatory of segregation due to transportation has not been fully developed; however, these observations have been made. When coning segregation during loading is minimized, segregation due to transportation minimizes, too. More segregation occurs during cone loading of the vehicle than in transportation.

Compaction decreases segregation by decreasing further movement of the particles. Compaction is maximum with the most orderly random arrangement of particles such as are attainable with spherical particles of equal diameter.

Segregation can be considered on a physical basis as well as a chemical basis. Physical segregation is the change in relative position of the particles from the primary homogeneous status, regarding only the size of the particle. Chemical segregation is the deviation of the chemical analysis of the "spot" sample from the original analysis of the blend. If the sample contains a considerable quantity of material, the analysis will show an average composition corresponding to the space from which it was taken. An excess in one component of the blend obviously implies a deficiency in another; if both of these blends are included in one sample, no segregation is found. As can be seen, the definition of average segregation depends on the amount of each component.

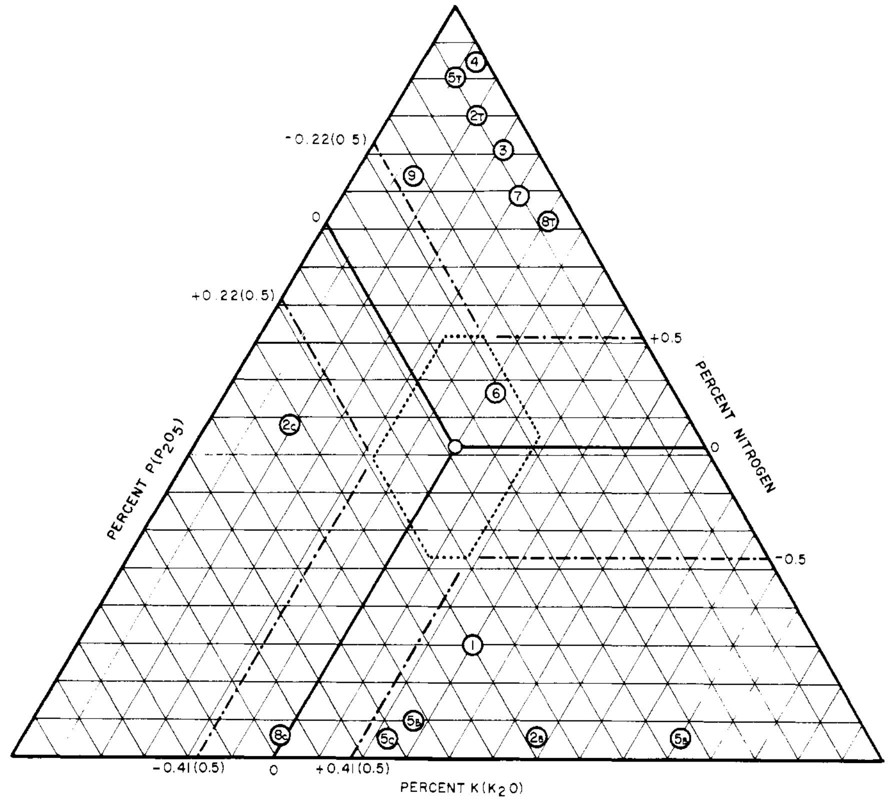

Figure 5. Chemical segregation of a blend of flaked ammonium nitrate, granular triple superphosphate, and coarse muriate of potash

Points inside the hexagon are within arbitrary chemical tolerances shown on the axes N, P, K

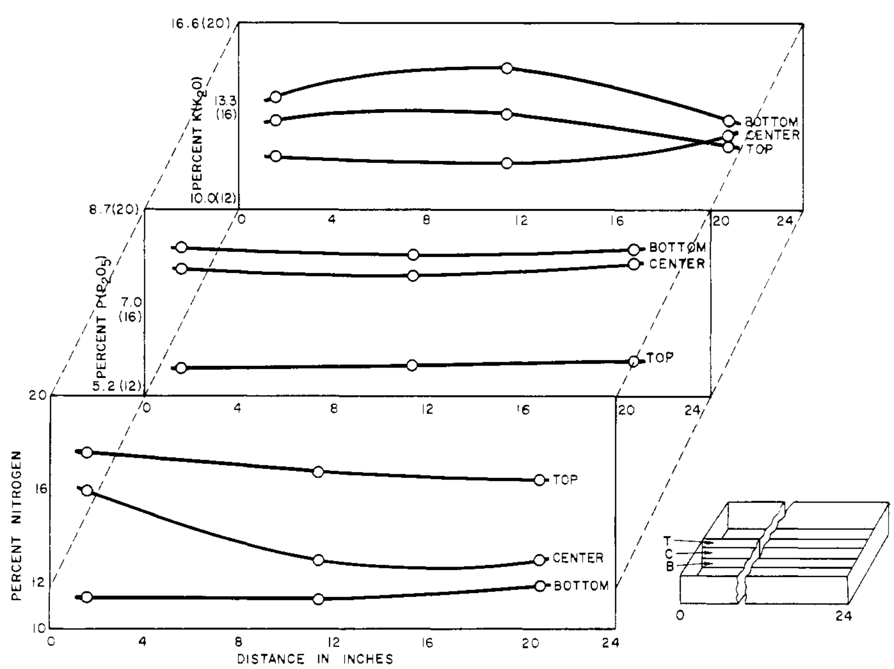

Figure 6. Chemical segregation of a blend of flaked ammonium nitrate, granular triple superphosphate, and coarse muriate of potash caused by vibration

In a mass, M, there is a sample, ΔM, whose analysis is p_1 with deviation from the original grade of Δp_1. If we analyze the entire mass, the number of samples is $\frac{M}{\Delta M} = n$, and the mean deviation is given by the formula:

$$\frac{1}{n} \sum_{1}^{n} (\Delta p_n)$$

This number is representative of the homogeneity of the mixture. As n increases, segregation will become more apparent; however, as the weight of the sample decreases, a point will be reached

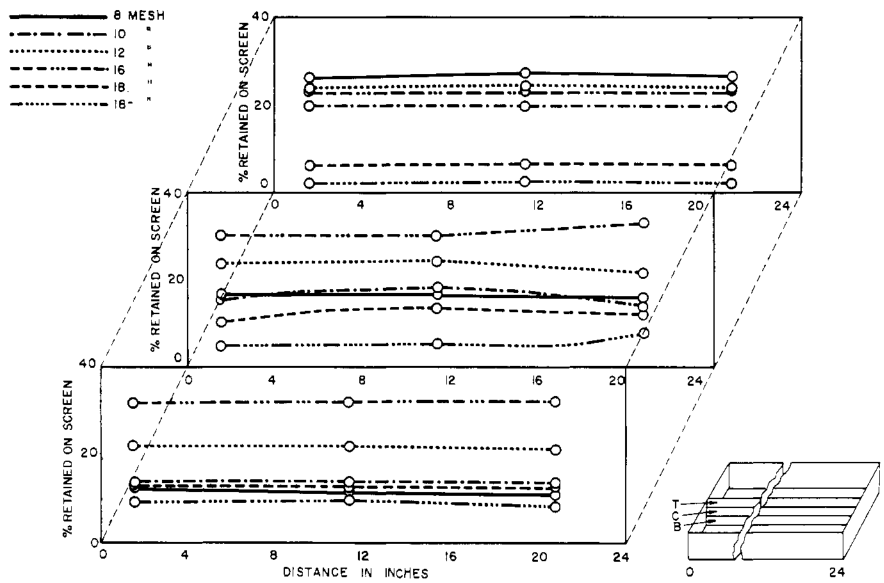

Figure 7. Physical segregation of a mixture of flaked ammonium nitrate, granular triple superphosphate, and coarse muriate of potash caused by vibration

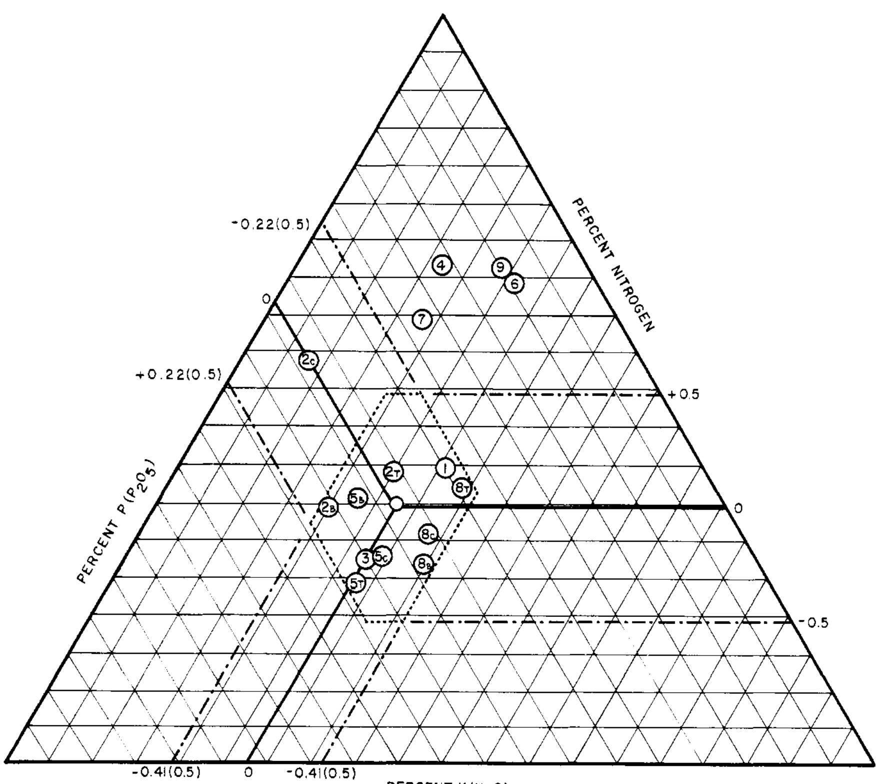

Figure 8. Chemical segregation in a blend of prilled ammonium nitrate, granular triple superphosphate, and coarse muriate of potash

Points within the hexagon are within arbitrary chemical tolerances shown on axes N, P, K

where the sample becomes so small that the random distribution of the particles produces bias, and the data cannot be applied to a study of the blend.

This study is for the purpose of improving the quality of bulk blended fertilizer; variables that in other industries represent serious problems (such as electrostatic charges) are considered negligible in this case. The segregation due to sifting of smaller particles through the voids between larger particles likewise has not been considered, because the usual size range of fertilizer raw materials for blending is adequately narrow. Hill (*7*) gives the approximate range of sizes to avoid this effect.

Variables Involved in Segregation

In a normal mixture of dry, blended fertilizers subject to external forces causing segregation, the following variables have been analyzed.

Specific Density of Raw Materials. The specific densities of the raw materials produced by different processes supplying our needs are so nearly equal that adjustments on this basis are not required. There is little choice and little need for selecting raw materials merely to obtain similar specific densities.

Bulk Density of Blend. An important variation in bulk density is observed in transportation segregation, where a reduction of volume occurs under vibration as the blend is transported. The moving particles reorganize themselves and compact to reduce further movement. Round particles have been found to be the best packing materials, probably forming a rhombic array; this can be an important factor in minimizing segregation.

Grade of Blend. Since it is the characteristics of the particles of the raw materials that affect segregation, the grade of blended fertilizer is of secondary importance.

Size of Particle. Particle size plays a very important role in segregation because friction factors are intimately related to size. Figure 4 shows friction factors as functions of size of particles for several materials widely used in the fertilizer industry.

Shape of Particle. As can be seen in Figure 4, the same material obtained by different processes has different shapes and different friction factor curves. Shape of the particles has a major influence on friction factors.

Possible Forces Acting upon Particles—Gravity, Friction, and Vibration. Gravitational force is a function of the mass of the particle; frictional resistive force is a function of the mass, surface rugosity, size, and shape. The physical mechanism of vibrating particles is complicated. Experimental observation of luminescent particles showed that the particles absorb vibration from the vehicle, the material compacts, and the top surface of the fertilizer becomes uneven.

In the authors' experimentation, two symmetric maximum levels or humps were found, the lower points of which exist in the center and close to the walls of the surface contour. Particles rise to the surface in the maximum level area and travel along the surface and toward the walls. As these particles reach the walls, they descend downward along it. The angle of the free surface in the vicinity of the wall depends on the intensity of vibration and on the material of which the wall is made. This has been clearly observed when measuring the junction angles for plastic and wood walls, for which the angles were considerably different. Of course, the wall effect will vary according to the shape of the vehicle in relation to the ratio of surface area to volume.

When the particles vibrate, the distance between them is greater than the particles can achieve by proper packing, the average distance between particles depending on the intensity of vibration. This vibration causes a gradient of bulk density with resulting undulations of the free surface of the fertilizer. In addition, the particles move with different friction factors, leading to the conclusion that the vibrating mixture in continuous movement approaches a gradient of concentration and size governed by the same relationships described for segregation produced in loading. In other words, the vibratory segregating factors are similar to those observed in loading.

The authors' analysis of segregation thus far shows that the friction factors are the controlling variables. Obviously, if two particles have the same friction factors and external forces, they will move at the same speed and direction, and the relative position will remain constant; in other words, under these conditions, there will be no segregation. Where each material is present in a range of sizes, segregation cannot be avoided completely, but it can be minimized by a proper choice of size range of the different ingredients of the blend.

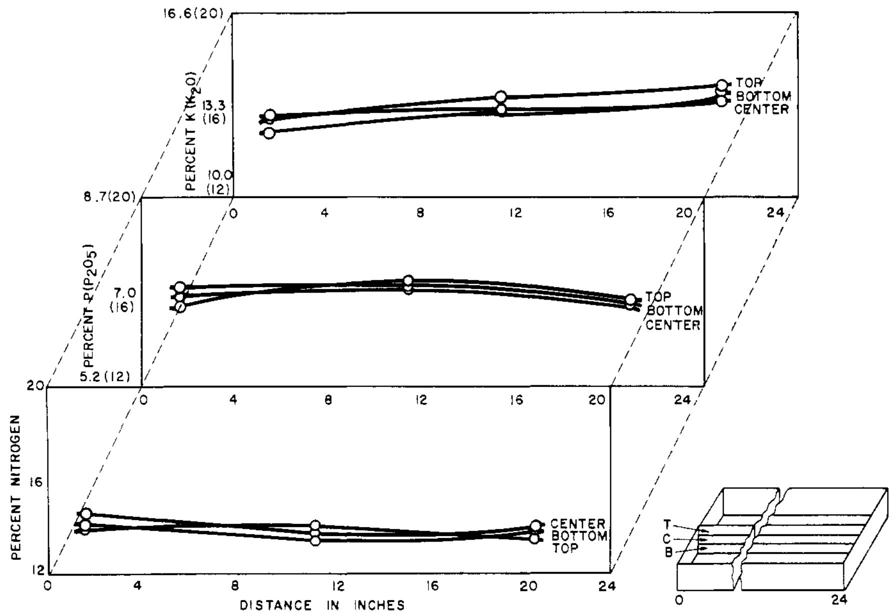

Figure 9. Chemical segregation of a blend of prilled ammonium nitrate, granular triple superphosphate, and coarse muriate of potash caused by vibration

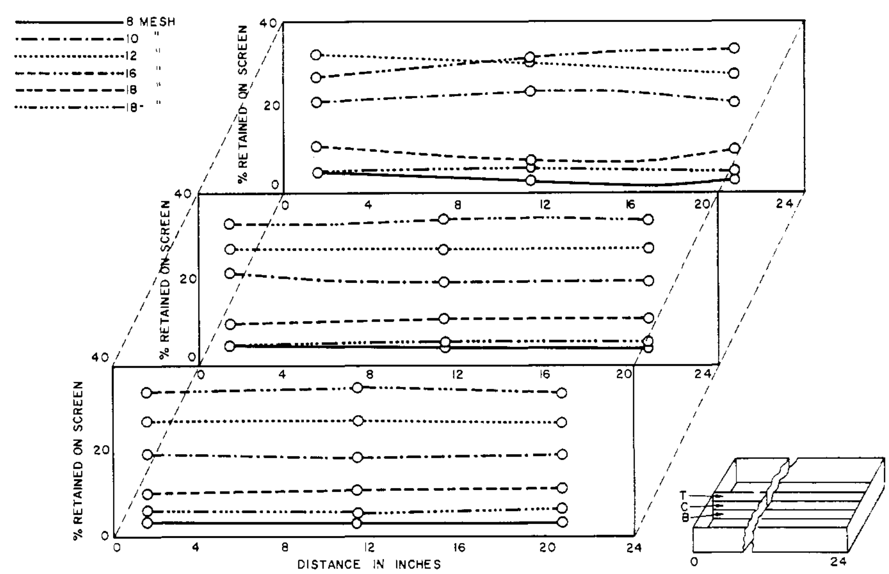

Figure 10. Physical segregation of a blend of prilled ammonium nitrate, granular triple superphosphate, and coarse muriate of potash caused by vibration

Experimental

Determination of Friction Factors. Chemical analyses were made according to A.O.A.C. procedures, and screen analyses were made with W. S. Tyler Co. equipment. When needed in conjunction with screen analysis of each component, the components of the mixture were separated by utilizing the differences in densities by floating in mixtures of tetrabromomethane or methylene iodide with carbon tetrachloride, and then each compound was analyzed separately. Trajectories of individual particles were observed by impregnating these particles with fluorescein and illuminating them with ultraviolet light.

To determine the friction factor curve for a given material, the material was separated into different screen sizes with the smallest size range possible. The material was then discharged through the bottom of a small hopper to make a cone, and the value of the angle of repose in the vertex was determined by photographing the pile. The value of the friction factor was determined by the relationship $\tan \alpha = \cot \beta/2 = k$ (Figure 3). Friction factor *vs.* size are plotted in Figure 4.

Plotting of Data. Chemical and physical segregation are illustrated in Figures 5 through 10. The triangular diagrams are used to show percentage segregation, since they are a simple way to represent in two dimensions a mixture of three components. Each vertex represents a pure compound; any point on one side represents a binary mixture, and any point inside represents a mixture of the three compounds. To simplify the plotting, the percentages of N, P (P_2O_5), and K (K_2O) have been substituted at the point representing the percentages of raw material ingredients, and the scale shown in the figure has been enlarged to magnify the separation between points. Around the point corresponding to the mixture before segregation, an irregular hexagon has been constructed to allow for a deviation of ± 0.5, ± 0.22, ± 0.41 in N-P-K. Points representing samples taken from the truck after the experiments are plotted on the same graph, enabling one to define a number that indicates the percentage of the over-all mixture that segregates. This number results from dividing the number of samples lying outside the hexagon by the total number of samples. It is best to think of this number as an index of segregation, and it is convenient as a method of comparing the tendencies of different mixtures to segregate.

Laboratory Tests. A laboratory testing method which will simulate in-truck segregation exactly has not been developed. On the other hand, field segregation testing cannot be as accurately and exhaustively conducted as in the laboratory; therefore, a compromise laboratory study was used to note the major variables involved in segregation. Due to the lower mass

Table I. Examples of Different Tendencies in Segregation

15–6–12 (15–15–15) blended of: (1) triple superphosphate, (2) ammonium nitrate, and (3) potassium chloride

Material and Shape	Size Range (Tyler Mesh)	Av. Friction Factor	Relative % Segregation
	TEST NO. 4		
(1) Granular	−8+18	0.65	93
(2) Flaked	−8+18	0.88	
(3) Coarse	−8+18	0.68	
	TEST NO. 5		
(1) Granular	−16+30	0.69	93.5
(2) Prilled	−8+30	0.68	
(3) Coarse	−16+30	0.60	
	TEST NO. 6		
(1) Compacted	−6+14	0.90	55
(2) Flaked	−6+14	0.88	
(3) Coarse	−6+10	0.82	
	TEST NO. 7		
(1) Granular	−8+18	0.65	33
(2) Prilled	−8+18	0.65	
(3) Coarse	−8+18	0.68	

retaining surface relationship, segregation was more acute because of container wall effect in the pilot plant than in the field for a given blend, with the probability that any mixture found to be satisfactory in these experiments will behave better in the field.

The capacity of the laboratory truck, which was built to scale from a full-size truck, is approximately 1 cubic foot, and the hopper and mixer are scaled accordingly. Calculated, preweighed, and mixed amounts of three ingredients were transferred from a loading hopper into the truck. The truck was vibrated in a manner designed to reproduce various road conditions for approximately 20 to 30 minutes to produce transportation effects. Samples were then taken at three points along the center $^1/_3$ of the truck representing three equal parts. Samples from the center of the truck supply maximum information because wall effects are eliminated. Each of the three samples in the center was then divided into three parts corresponding to the top, center, and bottom of the layer.

Results

Detection of Mixing Efficiency. The first step in preventing segregation is assuring mixing efficiency. Inefficient mixing may result from too short an interval in the mixer, improper mixer design, or improper feeding to the blender. This problem can be solved either by varying mixing time or by modifying the loading hopper. Obviously lack of proper mixing will be carried forward and added to normal segregation in the final nonhomogeneity.

Loading and Transportation Segregation. A method for predicting segregating tendency from an average friction factor for each of several raw plant food materials was developed. The average friction factor for a given material has been determined by multiplying the friction factor corresponding to each size by its percentage in screen analysis. The sum of all these values divided by 100 gives the averages tabulated in Table I.

Test No. 4 (Table I) shows that flaked ammonium nitrate has an average friction factor considerably higher than granular triple superphosphate and coarse potash, and high segregation should be expected with its use. In Test No. 5, triple and ammonium nitrate prills have about the same friction factors, but the potash is considerably lower. The analyses have shown that potash was the most segregated material of all three. Test No. 6 is an improvement of Test No. 4; the size of potash has been increased to increase the average friction factor. The range of size has been narrowed, and compacted triple has been used. The blend was very much improved. Test No. 7 has been made by choosing the size range of the components to obtain the same average friction factor. The result is a blend that was satisfactory under any handling condition simulated thus far. Two of the tests made are explained in detail to show techniques and the full information developed.

Comparison of the Segregation Tendency of Ammonium Nitrate in the Flake and Prill Forms when Combined with Granular Triple and Coarse Potash. To illustrate the correlation between average friction factor and segregating tendency in mixed fertilizers, experiments have been made with both flaked and prilled ammonium nitrate, keeping the particulate nature of phosphorus and potassium constant. Segregation was studied by taking vertical samples along the center line of the truck at three different levels, and overall samples on the sides. This procedure enabled comparison of the concentration of each component in the top, center, and bottom layers.

The grade of blend considered was 15–6–12 (15–15–15). Granular triple superphosphate and coarse potash were used in these test runs. The material was screened to −8+18 mesh with less than 5% of −18-mesh material allowed. In one portion of the test, ammonium nitrate prills were used, and in the other, flaked ammonium nitrate.

Calculated amounts of raw material were thoroughly mixed in the pilot mixer and loaded into a hopper and then into a truck by a procedure designed to minimize loading segregation. After being loaded, the truck was left on the shaker for 2 to 3 hours. The truck was then sampled in accordance with the pattern mentioned. Samples in the center were divided into three horizontal sections, and all samples were subjected to physical and chemical analyses. The analytical data obtained from the samples described in the previous section are plotted in Figures 5 to 10. These results should not be construed to mean that flaked or compacted materials should not be considered for physical blends; the results merely show that materials whose k curves differ should be sized so that average friction factors are as close to equal as possible.

Flaked Nitrate. CHEMICAL TEST. Figure 6 shows how the three compounds are rearranged in definite layers to give a very high segregation. From top to bottom, the concentration of ammonium nitrate decreases rapidly. The concentration of K (K_2O) and P (P_2O_5) increases from top to bottom. There is a strong segregating tendency, especially between the flaked ammonium nitrate whose average friction factor differs from that of the other two components and coarse potash, that made these two materials incompatible for this blend. In Figure 5 are plotted the chemical analyses of each sample, and only one out of 15 samples lies inside the permitted zone. This gives an index of segregation of $\frac{14}{15} \times 100 = 93.5\%$.

PHYSICAL TEST (FIGURE 7). The marked segregation of the 8-mesh particles is illustrated in Figure 7, where the line representing the 8-mesh material varies from top to bottom. The line representing the 10-mesh material does not drop as much, and the drop of the 12-mesh line is negligible. The 16-mesh material shifts in the opposite direction, as does 18- and −18-mesh material to an even greater extent. The size of particles that do not shift is about 14 mesh.

Prills Run. CHEMICAL TEST. Figure 9 represents a blend of low segregating tendencies. Probably a structure close to a rhombic array has been produced by the ammonium nitrate prill. The triangular representation (Figure 8) shows only $\frac{5}{15} \times 100 = 33.3\%$ segregation.

PHYSICAL TESTS (FIGURE 10). The exchanges produced in the different size concentrations are negligible, except in the top part of the center line, where some small distortions have been found. The physical analysis indicates good compaction in the material and very small movement of the particles. The friction factors are very close (Table I, test 7).

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