CONTINUOUS MIXING OF SOLIDS

(met een samenvatting in het Nederlands)

by/*door*

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INTRODUCTION AND SCOPE OF THE STUDY

Traditionally, the physical sciences because of their earlier development have been used to provide the theoretical background for the different unit operations. In general, it can be said that the solid state of matter was and still is more difficult to subject to scientific investigation than the fluid state. Industrial processes, which by their very nature have to deal with physical phenomena, are by far better described scientifically in the case of fluids than in the case of solids. ROSE (1960), in a recent article, concluded that the present knowledge about such apparently simple solid properties as roughness, stickiness and electrical properties all of which affect the technical behaviour of particulate materials is far from satisfactory. In spite of these facts, the processing of materials in the solid state is of very wide occurrence in industry. Due to the lack of systematic knowledge, many industrial solids processing methods necessarily had to follow an empirical path in their development.

Solids-solids mixing is a notable example of a widely used unit operation in industry which was developed as an empirical art without fundamental theory. Processes developed purely along empirical lines generally are not optimal in performance and may result in appreciable losses of power and material. Because of its importance to industry, batch solids mixing has received greater attention from different workers during the last several decades and attempts have been and still are being made to establish a scientific foundation for this process.

Up till now, developments in the theory of solids mixing have been limited mainly to the field of batch mixing. Pioneering work in this field seems to have been started by OYAMA in 1933 [see WEIDENBAUM (1958)]. Among other things, OYAMA studied and defined the state of motion of granular material in a horizontal rotating cylinder and successfully used a photographic method for following the course of a mixing operation.

In his earlier studies, OYAMA definitely approached the mixing process from the point of view of the dynamics of particles in a mixer. He [OYAMA (1940)] considered mixing as "the operation causing mutual movement of particles of matter for the purpose of bringing them to the state of complete mixing". Citing the work of KIELLEFER (1923), OYAMA in the same article stated "the motions by which this operation will be accomplished in practice may be roughly classified into eight groups: free rotational motion, impeded motion, tumbler motion, shaking motion, ball mill motion, straight line motion, and random motion". The earlier work of OYAMA dealt with several types of the motions mentioned.

Real progress in batch solids mixing theory only took place after LACEY (1943) described the mixing process in terms of a simple statistical model. According to this model, which will be described in greater detail later, the end result of a mixing operation is not a systematic distribution, but it should be a random distribution of particles which can be analysed by statistical methods.

In practice solids are frequently mixed in relatively small batches and there is no doubt that the greater attention which batch mixing received from the different investigators can be attributed to this fact. On the other hand, especially in the Food and Agricultural Industries, it is often necessary to process very large volumes of solids.

There is probably an upper limit to the size at which batch mixers can be built and still at the same time function economically and in an efficient way. For mixing large batches of solids circulating systems or large tumbling barrels have been used [VALENTINE and MACLEAN (1950)]. Circulating systems represent a mechanical quartering method and therefore are slow. Large tumbling barrels give incomplete mixing unless excessive time is consumed because they do not produce good end- to end flow of material, even when provided with internal baffles.

Another arrangement frequently encountered for mixing large batches of solids in industry, is to use several batch mixers interchangeably. While one mixer is emptying its charge, a second is mixing and a third is being filled, so that a continuous outflow of product is maintained. It is obvious, however, that such a system, which works rather well in practice, does not represent the most economical method of mixing large quantities of solids.

Continuous mixing raises no limitations as to the quantity of solids to be processed. Also when forming an intermediate operation in a manufacturing process, as is usually the case, continuous mixing would not require large intermediate storages as would be the case when batch mixing is applied. Furthermore, it is more economical to maintain process equipment in continuous and steady operation, with a minimum of disturbances and shutdowns. Such advantages together with the fact that feed regulation can now be easily performed electronically will undoubtedly encourage a shift to take place from batch to continuous operation. That this is actually happening at the present becomes evident from the fact that several manufacturers are trying to adapt their batch mixers to continuous operation by joining several of these mixers in series and allowing the feed to enter continuously from one side and to leave continuously from the other side of the series of mixers.

During the last decade or so much theoretical work has been devoted to continuous processes in general and continuous fluid flow blenders in particular. Unfortunately, most of the published work cannot be applied to the study of continuous solids mixing, since the greater part of the available theory is definitely limited to certain specific problems such as the smoothing-out of fluctuations in feed streams by continuous flow blenders, or the determination of the efficiency of chemical reactors [e.g. BEAUDRY (1948), DANCKWERTS and SELLERS (1951), DANCKWERTS (1953b), KLINKENBERG and SJENITZER (1956), KATZ (1958) and DE BAUN and KATZ (1961)]. Still some of the theoretical aspects developed in this field of investigation, in particular the concept of using the holding or residence time distribution for the characterization of continuous flow equipment, can find application in experimental studies dealing with continuous solids mixing. The basic ideas involved here will be reviewed at another place.

At this point it is only necessary to draw attention to the fact that continuous solids mixing in its true sense has, as far as we know, not as yet been investigated. The reason for this may be that such investigations are extremely difficult and laborious.

The present study was undertaken as a start in this direction. Our study is limited to particulate solid materials which can be called coarse grained. Mixing fine powders and also the mixing of two or more materials with different physical properties have not been studied. Two continuous mixers employing different mixing mechanisms have been used. The first is a pilot

plant model of an industrial continuous solids mixer manufactured by the SPAANS MACHINE FABRIEK, Hoofddorp, Holland, and the second is a purely experimental mixer of a rotary type, designed and constructed at the LABORA- TORY OF AGRICULTURAL TECHNOLOGY in Wageningen.

In chapter 2 the basic principles of the theories of batch solids mixing and continuous-flow systems which bear some relation to the present investigation will be briefly reviewed, for the purpose of clarifying the reasons for the choice of methods and procedures used in this study. In chapters 3 and 4 we discuss the investigations carried out on the two mixers, and in chapter 5 we conclude with a general discussion.

<div align="center">CHAPTER 2</div>

THEORETICAL ASPECTS OF MIXING OF SOLIDS

In this chapter we will give a general description of the mixing of solids and discuss various theoretical aspects of the process with the aim to lay a foundation for the experiments to be described in the following chapters and for the treatment of the results thereof. At the same time we will review the most important and relevant literature. As will be seen below by far the most publications deal with batch mixing; however the theory of this process can also be applied, at least partly, to continuous mixing. Successively the mechanism of the process, the possibilities of defining and determining a degree of mixing, the phenomenon of segregation and the rate of mixing will be discussed; finally some specific problems occurring with continuous mixing, as the distribution of residence times, will be dealt with briefly.

Solids mixing theory deals with the qualitative and quantitative description of the processes taking place in a mixer and the end result of these processes. Although a mixer of solids is a relatively simple machine and the mixing of solids itself is an apparently simple operation, yet the processes taking place in a mixer are so complex that it is very difficult to treat them analytically. LACEY (1954) described the *mixing mechanisms* in terms of the processes of convection, diffusion and shear. According to him, (*i*) convective mixing refers to the transfer of groups of adjacent particles from one location in the mass to another, (*ii*) diffusive mixing is the distribution of particles over a freshly developed surface, and (*iii*) shear mixing takes place when slipping planes within the mass occur. By far, the diffusion analogy has been the most used in solids mixing theory.

It must be realized, however, that because of the complexity of the process any hypothesis which attempts to describe it can only be considered as a very rough approximation to the actual facts. Certainly no single description can cover all cases, which are many and varied, although for a certain machine operating under certain conditions a hypothesis can be in good agreement with the actual facts.

Leaving the mechanisms of the process out of consideration, an important aspect of solids mixing theory deals with the quantitative methods of *defining and measuring the degree of mixing*. Such methods are required for specifying mixing tasks and for comparing the effectiveness of mixing processes.

At this point it is necessary to differentiate between two general types of

mixtures. For this purpose it is appropriate to introduce the concept of the "scale of scrutiny" as proposed by DANCKWERTS (1953a). According to DANCKWERTS, it is impossible to decide whether a mixture is well or badly mixed unless the reasons for making it up are known. Any mixture, if scrutinized closely enough, will show regions of segregation, that is the composition will vary from point to point. The size of the regions of segregation which can be tolerated will vary from one case to another. DANCKWERTS applied the term "scale of scrutiny" to the minimum size of the regions of segregation in the mixture which could cause it to be regarded as imperfectly mixed for a specified purpose.

The components of any mixture consist of particles which are capable of relative movement. These particles may be large or small. If the scale of scrutiny is not large enough to embrace many particles, DANCKWERTS called the mixture "coarse-grained", but when the scale of scrutiny is great enough to embrace a very large number of particles of each component, the mixture was called "fine-grained". These two types of mixtures differ with respect to the most appropriate methods of investigating them, because the fluctuations in composition which are obtrusive in coarse-grained mixtures become unimportant in fine-grained mixtures.

The scale of scrutiny or more simply the practical size of a sample to use under any set of conditions for analysing the quality of a mixture is thus determined by the later use of the mixture. As far as solids mixing theory is concerned fine-grained mixtures in the sense just defined would most probably be limited to the case of very fine powders and although DANCKWERTS (1952) has given measures for studying them, such measures require measurements on point concentrations which are definitely very difficult to perform on solids. Solids mixing theory is thus mainly directed to coarse-grained mixtures, and is based on statistical considerations which we shall discuss. HEMELRIJK (1954) has shown that these statistical methods can also be used for the analysis of samples containing an unknown large number of particles.

The starting point is the *statistical model* describing the mixing process. This was originally suggested by LACEY (1943), but we shall describe it here in more general terms. The model is based on the statistical concept of randomness. Two general cases will be discussed depending on the presence or absence of factors which act to oppose complete randomisation of the particles. We shall first deal with the latter case.

In its simplest form the model assumes the absence of segregating effects. For coarse solid particles this means that in a gravitational field, the particles to be mixed are indistinguishable as far as gravitational and inertial forces are concerned. They have the same shapes, sizes and densities and differ only in a gravitationally neutral property such as colour. For fine powders, roughness, stickiness and electrical properties are further examples of factors which can give rise to segregation.

On the consequences of the existence of segregation processes and on the possibility to allow for these phenomena in the mathematical model of the mixing process we shall come back later.

Under the assumption that segregating effects are absent, the beginning and the end of a mixing process can be defined by two limiting "arrangements" of particles. At the start, the particles of the different components are completely separated but meet at the interface. They are orderly arranged in heaps or

layers and in this form represent the first limiting arrangement of particles; namely, the "completely segregated arrangement". This is a very special arrangement.

Mixing brings the particles into a state of motion. The created movement allows the particles to exchange positions. The system moves away from the very special arrangement to one (any) of the extremely large number of non-distinguishable nonspecial arrangements. These all together are covered by the name "random arrangement". This latter is now considered to represent the optimum end result of the mixing process.

In any mixing operation a great many different arrangements of particles will occur between the initial segregated arrangement and the final random arrangement. They are distinguished by the variability of some sample property which has a maximum value for a segregated arrangement and a minimum value for a random arrangement. In between these two extremes the variability will be dependent on the time of sampling, since it continuously decreases from the maximum to the minimal value.

A mixture of two (or more) kinds of particles will be considered as a finite set of places at each of which one particle is situated. A random mixture is such that all arrangements of say p particles of the first kind and q of the other kind have the same probability. The chance to find at a given place a particle of the first kind is $p/(p + q)$. This type of distribution is called a Binomial probability distribution.

Generally, the distribution of one kind of particles in a mixture is characterized by the known (P) or the experimentally evaluated ($\bar{x}$) *mean proportion* of the kind of particles considered and by the *variance* of the proportions found in an infinite number of samples of a definite size, which variance is more or less accurately estimated from a higher or lower finite number of samples. Where the mean composition is a constant, the variance is a function and thereby a criterion for the degree of mixedness. We shall discuss now the suitability of the variance of sample compositions as a measure for mixture quality and state theoretical maximum and minimum values of it.

The variance is estimated in the following way. If x_i is taken to represent the proportion of the component under consideration in the ith sample, then the experimental variance s^2 can have either of the following two forms depending on whether P or $\bar{x}$ is used:

1. $s^2 = \sum_{i=1}^{N} (x_i - P)^2/N$ with N degrees of freedom.

2. $s^2 = \sum_{i=1}^{N} (x_i - \bar{x})^2/N - 1$ with $N - 1$ degrees of freedom.

The variance as a measure of mixture quality has its limitations. The main objection against using the variance as a measure of mixture quality is its dependence on sample size. For simple cases met in practice, where it is only required to conform with certain specifications, the dependence on sample size can be corrected for by relating the variance to its theoretical minimum value in the form of a "mixing index". As we shall see later even this correction will not be sufficient under certain conditions.

As a *theoretical maximum* value for the variance (σ_0^2), LACEY (1954) proposed using $P \cdot Q$ where $Q = (1 - P)$ on the condition that samples partly covering

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the interface can be neglected. In practice this value will overestimate the true variance at zero time. The deviation between the two values will be greater as the number of interfaces increases. An experimental value for the variance at the start of mixing is therefore preferable to the theoretical value $P \cdot Q$.

Concerning the *theoretical minimum* value of the variance, which is denoted by σ_r^2, several forms have been suggested. LACEY (1943) considered the simple case when two components are mixed in the absence of factors which act to oppose complete randomisation. In such a case the minimum value of the variance can simply be represented by the variance of a Binomial distribution which is equal to $P \cdot Q/n$, where $n =$ the number of particles per sample. On the other hand, BLUMBERG and MARITZ (1953) preferred to express this minimum value in terms of a transformed distribution of sample proportions. They do not, however, give proper reasons as to why they introduce the concept of transformation to mixing theory. They simply state: "The calculations are simplified if the variables x_i are transformed to new variables z_i according to $z_i = 2 arcsin \sqrt{\bar{x}_i}$".

In statistical science transformations are, however, used to partly satisfy the assumptions underlying an analysis of variance, namely for normalising the data and equalising the variance. When the original variate x has a distribution for which the expectation (mean) is $E(x) = m$ and the variance is Var.$(x) = f(m)$, and $f(m)$ is not constant, it is necessary to find a new variable z, a function of x, for which the variance will be independent (or nearly independent) of m, so that the necessary assumptions required for an analysis of variance are satisfied.

For a Binomial distribution the variance is a function of the mean and thus the use of transformations for such a distribution becomes relevant. The appropriate function for the Binomial case is the angular transformation, $arcsin \sqrt{x}$. This transformation has a fairly constant variance equal to $1/4n$ or $820.7/n$ depending on whether the angular value is taken in radians or degrees, respectively. Since BLUMBERG and MARITZ (1953) used $2 arcsin \sqrt{x}$ instead of $arcsin \sqrt{x}$, the minimum theoretical value of the variance for this case becomes $1/n$, where again $n =$ number of particles per sample.

Because in practice the particles which are mixed are not usually uniform in size, the problem of defining a σ_r^2 for mixtures of multi-sized particles assumes great practical importance. Such particles are, however, distinguishable in a gravitational field and are thus subject to segregation. Furthermore, a true random distribution of the individual particles of the different sizes can never be achieved, because the smaller particles tend to pack in the voids existing between the larger ones. In spite of these facts, MANNING (1937), BUSLIK (1950), STANGE (1954) and HYUN and DE CHAZAL (1962) attempted to define a σ_r^2 for such mixtures.

MANNING (1937) tried to apply the variance of a Binomial distribution to the largest size of a multi-sized mixture by expressing its content in the mixture on the basis of weight fraction instead of particle numbers, but without success. STANGE (1954) derived a formula on the basis that particles of different sizes can still be randomly distributed in the mixture. Because of the reasons given above, the validity of this assumption becomes very doubtful and accordingly it is difficult to draw any conclusions concerning the conditions under which his formula will hold in practice.

BUSLIK (1950) modified MANNING's method by considering each size com-

ponent as being binomially distributed throughout the volume of the mixture. His formula, however, only gave good results for the large size of particles but was in poor agreement for the smaller sizes. HYUN and DE CHAZAL (1962) attributed the failure of BUSLIK's formula to the existence of gaps between large particles, which BUSLIK's model does not take into consideration. According to them, these gaps can only be filled by small particles and at the same time a certain amount of the volume of the mixture becomes unavailable for the medium size particles. They refer to the latter as the "excluded volume". Basing upon this model they developed expressions for σ_r^2 for large, medium and small particles, and state that although their method may be extended to any number of particle sizes, its complexity increases so rapidly as to make it unwieldy.

HYUN and DE CHAZAL reported the results of three trials to show that their correlation gave better agreement with the experimental results than that of BUSLIK. They rightly indicate, however, that the agreement is not exact and attribute the discrepancy between the theoretical and experimental values to the limited number of the samples used in their trials. It is clear therefore that more experiments are required in order to test the range of applicability of their proposed equations.

The variance of sample composition is a measure of mixture quality only in its comparison with its theoretical minimum and maximum values. Several definitions of a *mixing index*, i.e. a measure of the state of mixedness, based on the comparison of experimental with theoretical variances have been proposed and some of them will be discussed here.

According to HERDAN (1960) the comparison of an observed standard deviation with the theoretical value was first introduced by LEXIS (1877) for the purpose of ascertaining whether a universe from which samples had been taken was homogeneous, or whether the probability of the event in question differed significantly from place to place, or from time to time. In industry the test of the hypothesis that the population variance is equal to a given value, is extensively used as a method of quality control. Irrespective of these facts and as far as mixing theory is concerned the concept was not seriously considered until LACEY suggested it again in 1943.

There are several possible ways of making the comparison. If M is taken to indicate a mixing index, then $M = \sigma_r/s$ was one of the simple relations first suggested by LACEY (1943). Other simple forms of M have been used by MICHAELS and PUZINAUSKAS (1954), SMITH (1955) and SAKAINO (1956).

KRAMERS in a private communication to LACEY (1954) suggested using $M = (\sigma_0 - s)/(\sigma_0 - \sigma_r)$. Because the variance, unlike the standard deviation has additive properties, LACEY (1954) replaced the standard deviation by the variance and further developed the expression to:

$$1 - M = (s^2 - \sigma_r^2)/(\sigma_0^2 - \sigma_r^2).$$

The sampling properties of this index have been discussed by LACEY (1954). For diffusive mixing, the index is independant of sample size, but for convective mixing which involves the transfer of groups of adjacent particles from one location in the mass to another, M will depend very greatly on sample size.

From the statistical point of view a test for completeness of mixing is usually based on formulating the null hypothesis that a set of sample means are equal. This hypothesis is then simply tested against the alternative hypothesis that

these sample means are not equal. When sampling from a Binomial population the statistic to use for testing the null hypothesis that N population means are equal is actually the Chi Square (χ^2), which like the variance can have one of the two following forms:

$$\chi^2_N = \frac{n \sum\limits_{i=1}^{N} (x_i - P)^2}{P \cdot Q} \qquad \text{or} \qquad \chi^2_{N-1} = \frac{n \sum\limits_{i=1}^{N} (x_i - \bar{x})^2}{x(1 - \bar{x})}$$

Here n = number of particles per sample. This is assumed to be constant, but when this is not the case, either an average value can be taken or each squared deviation is first multiplied by the number of particles of the sample before the summation is carried out.

If the Chi Square is divided by the number of degrees of freedom $(d.f.)$ then we actually are comparing variances. It can then be easily seen that $M = \sigma_r/s$ is equal to $(d.f./\chi^2)^{\frac{1}{2}}$. On the other hand BLUMBERG and MARITZ (1953) preferred to use χ^2 itself as a measure of the state of mixedness after the data was transformed to $2\,arcsin\,\sqrt{\bar{x}}$.

In principle the statistical hypothesis that N population means are equal, can be tested by an analysis of variance or by means of the Chi Square test as described above.

In mixing studies the latter method would be used by preference, since the problem involves a simple design of complete randomisation and not a complex experimental design in which case the analysis of variance would have to be used. Now the only statistical assumption underlying a Chi Square test is that the samples are random [LI (1961)]. There are no assumptions regarding the variances as is the case with an analysis of variance and accordingly the usage of transformations in a Chi Square test seems unwarranted. According to LACEY (1954), however, the angular transformation renders a mixing index independent of P, although still the correction is considered to be of theoretical importance only. On the other hand, HEMELRIJK (1954) stated that BLUMBERG and MARITZ only complicate things by needlessly applying the angular transformation.

The Binomial distribution is a dichotomous distribution and strictly speaking can only be applied to two component systems. Its application can be extended to the case of multicomponent systems by considering one of the materials to be mixed as one component and all other materials as a second component. Specific statistical methods are, however, also available for the analysis of multinomial distributions and were introduced to mixing theory by HEMELRIJK (1954) and GAYLE, LACEY and GARY (1958). The statistic to use in testing the hypothesis that N samples are drawn from the same r-categoried multinomial population is again (χ^2) but in the following form:

$$\chi^2 = \sum^{Nr} \frac{(O - E)^2}{E}$$

Where O is an observed frequency and E the hypothetical frequency. For large samples, this statistic follows the Chi Square distribution with $(N-1)$ $(r-1)$ degrees of freedom. As a mixing index GAYLE and coworkers (1958) suggested:

$$M = \frac{\chi_o{}^2 - \chi_r{}^2}{\chi_s{}^2 - \chi_r{}^2}$$

where $\chi_o{}^2$ = observed Chi Square for any mixture
$\chi_r{}^2$ = expected Chi Square for a random mixture
$\chi_s{}^2$ = expected Chi Square for a segregated mixture.

In a more recent article GAYLE and GARY (1960) suggested using an experimental value for $\chi_s{}^2$.

HEMELRIJK (1954) drew attention to the fact that the best sampling plan to be adopted depends on the alternative hypothesis against which one wishes to test the hypothesis of random mixing. The place of sampling, testing for the presence of special trends in the mixture or testing for the presence of lumps (clotting or clustering) are all examples of alternative hypotheses against which the hypothesis of random mixing can be tested.

This was actually done by SHINAR and NAOR (1961). They suggested two major alternatives to the null hypothesis formulated for testing the completeness of mixing; namely:

A– Existence of a large scale gradient of concentration.
B– Appearance of local clusters which may be distributed at random.

According to them the classical Chi Square test for completeness of mixing would only apply to case A, since the existence of a gradient is associated with an increase in variance which may be calculated. In case B, the presence of clusters would not only render the variance dependent on sample size but the increase in variance would be comparatively small and hard to detect by the Chi Square test. They further argue that although the Chi Square test would be sufficient for many practical investigations, there are some processes which require a maximum degree of randomness and it is therefore necessary to have a test sensitive for the detection of clusters.

Accordingly they developed a test which is based on the shortest distance between particles. They reason that the shortest distance between particles is suitable for the purpose of the test because when clustering occurs the particles would be less widely spaced in some regions and moreover the shortest distance between particles is a measurable variable with the desirable property of being independent of sample size and form. The test involves transforming the distribution of N independent squared shortest distances into a χ^2 – distribution with $2\ N$ degrees of freedom according to

$$\chi_{2N}^2 = 2\ \pi\mu \sum_{i=1}^{N} r_i{}^2$$

where $r_i{}^2$ = the ith squared shortest distance between two particles.
μ = the number of particles of component under consideration per unit of tested area.
π = numerical constant.

The hypothesis of randomness is rejected for values of $2\pi\mu \sum_{i=1}^{N} r_i{}^2$ smaller than the Chi Square value with $2N$ degrees of freedom at the chosen probability level of significance.

For practical application, the authors draw attention to several drawbacks of the method. Apart from the considerable amount of work required for measuring and calculating the data, it is limited to those cases where one of the ingredients is present in quantities less than five percent and where the particles are uniform in size and are distinguishable by some optical method.

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If the materials to be mixed differ widely in physical properties, a *"segregation process"* might counteract the "mixing process". Apparently an equilibrium state of incomplete randomness of component distribution is arrived at, if the rates of the two processes are equal but in opposite directions. DOORNBOS (1956) gave a statistical test against gravitational segregation (i.e. a trend in the amount of one component in a vertical direction) which is based on the coefficient of rank correlation of SPEARMAN.

Segregation phenomena can make all the difference between the success or failure of a mixing operation; but in spite of such importance they have not received much attention in mixing theory until recently. For coarse loose dry materials, an attempt has been made by WILLEMSE (1961) to characterize the intensity of segregation by means of a dimensionless grouping in the form of a Peclet number. Also ROSE (1959) and WEYDANZ (1960) have developed rate equations for the simultaneous occurrence of the two opposing processes of mixing and segregation.

Solids mixing rate theory has been dealt with by several workers, as examples the work of BROTHMAN et al (1945), COULSON and MAITRA (1950), LACEY (1954) WEIDENBAUM and BONILLA (1955), OYAMA and AYAKI (1956), ROSE (1959) and WEYDANZ (1960) can be cited.

The initial approaches to the problem did not take into consideration the process of demixing and usually involved some hypothesis which lead to a differential equation of the following form:

$$\frac{d\nu}{dt} = k\,(\nu - \nu_{eq}).$$

where ν = some measure of mixture quality
ν_{eq} = the equilibrium value of the measure of mixture quality
t = time of mixing
and k = a constant

Such a simple differential equation has been integrated to give equations of the type:

$$M = 1 - e^{-kt}$$

in which M represents some form of mixing index, t is the time of mixing and k is taken to represent the rate constant of the process and thus to be dependent on the physical characteristics of the material being mixed as well as on the geometry of the mixing machine.

Basing on a diffusion analogy, LACEY (1954) applied FICK's law of diffusion to the horizontal mixing taking place in a horizontal rotary cylinder, and represented the mixing rate by a diffusion coefficient. His derivation is thus not applicable to the more practical case of radial mixing. This is usually considered to be very fast in batch mixers.

ROSE (1959) however proposed representing the process by a differential equation which was formulated on the basis that a "mixing potential" and a "demixing potential" are operative.

According to him the rate of mixing can be represented by:

$$\frac{dM}{dt} = A\,(1 - M)$$

and that of demixing by:

$$\frac{dM}{dt} = B\emptyset$$

so that the differential equation of the whole process becomes:

$$\frac{dM}{dt} = A(1 - M) - B\emptyset$$

Here, $M = s/\sigma_0 = $ a mixing index, $t = $ time, $\emptyset = $ a variable dependent upon the distribution of the components in the mixer, $A = $ a constant dependent upon the geometrical and physical properties of the mixer and the materials, upon which the rate of mixing depends and $B = $ a constant dependent on the physical properties of the materials mixed.

Because the "demixing potential" can be either positive or negative, depending upon the distribution of the components in the system, the solution of the proposed differential equation resulted in the following two equations:

$\emptyset + ve$
$$M = 1 - [(1 - B/A) e^{-At/2} + B/A]^2$$

$\emptyset - ve$
$$M = 1 - [B/A - (1 + B/A)e^{-At/2}]^2$$

ROSE further states that although these two equations explain the behaviour of the material in a mixer with fair precision they are not completely satisfactory for application to the whole process, because although the first equation leads to a maximum value of $M = 1$, test results upon real mixers suggest that no real machine leads to a perfect mixture. Accordingly he found it necessary to introduce an "intrinsic efficiency" of the mixer, η, into the equations; this "intrinsic efficiency" being a measure of the perfection of the machine as a mixing device and independent of the nature of the charge of the mixer. Thus the final forms of the equations become:

$$M = \eta \{1 - [(1 - B/A)e^{-At/2} + B/A)]^2\}$$

$$\text{or} \quad M = \eta \{1 - [B/A - (1 + B/A)e^{-At/2}]^2\}$$

Another notable effort to extend the initial simple differential equation to cover more complex cases was made by WEYDANZ (1960) who considered the simultaneous occurrence of mixing and unmixing movements. According to him the processes in a mixer are so complex that it is impossible to represent them exactly in detail in a manageable mathematical form. It is necessary to find a simplified model, that can be easily described mathematically and notwithstanding its deviation from reality gives a useful agreement between theory and experiment.

He defines the mixing movements as the exchange of places between volume elements with high concentrations with others with low concentrations so that the concentration differences are smoothed out. On the other hand demixing is considered to occur when the particles of one component obey other laws in their movements than the particles of the other components so that there results a preference for the introduction of these particles to certain places. In any machine the mixing and unmixing movements should be separately treated depending on whether a vertical or a horizontal direction is considered. In a vertical direction there is a mixing movement with an unmixing movement

superimposed on it. On the other hand in a horizontal direction no unmixing and only a mixing process is operative.

By considering the volume to be divided into four parts, namely upper and lower, and right and left, WEYDANZ represented the processes of mixing and unmixing by a set of differential equations representing the exchange of volume elements between the postulated parts. The solutions of these equations were then related to the standard deviation by assuming that if sampling were uniformly performed throughout the mixture, then the number of samples with a certain concentration of a certain component is proportional to the relative volume of that component in the mixture. In this way he arrived at the following relation which represents the overall processes of mixing and unmixing taking place simultaneously in the vertical and horizontal directions:

$$\sigma = \frac{s}{s_0} = \sqrt{e^{-2at/y \cdot (1-y)} + \left(\frac{0.5\ c}{s_0 \cdot b}\right)^2 \cdot (1 - e^{-4bt})^2} =$$

$$\sigma = \sqrt{e^{-2At} + \sigma_\infty^2 (1 - e^{-BAt})^2}$$

σ = mixing index which is referred to as a reduced standard deviation
σ_∞ = limiting value of σ at $t = \infty$ and is equal to s_∞/s_0
y = volume fraction of component considered, in whole mixture
s = overall standard deviation for the mixing and unmixing processes
s_0 = limiting value of s at $t = 0$ and is equal to $\sqrt{y \cdot (1-y)}$
s_∞ = limiting value of s at $t = \infty$ and is equal to $0.5\ c/b$
a = a constant representing the mixing intensity in the horizontal direction.
b = a constant representing the mixing intensity in the vertical direction
c = a constant representing the unmixing intensity in the vertical direction
t = time of mixing
A = a/s_0^2
B = $4 \cdot s_0^2 . b/a$

Although these two equations can be made to fit the experimental results very well and are actually a tremendous improvement to the earlier equations developed for this purpose, they are still subject to several objections. In the first place a close fit does not necessarily mean that the theory applies because of the large number of constants used. Furthermore some of these constants are either very difficult to determine like the B in WEYDANZ's equation or they do not find a place in the theory like ROSE's intrinsic efficiency. The most important objection is, however, that their mixing indexes are dependent on sample size. Since the equations have been developed for the case where segregation takes place, a minimum value for the variance could not be defined theoretically. Accordingly its maximum value σ_0^2 (in our notation) was used and this unlike σ_r^2 cannot correct for sample size. Their usefulness for comparative purposes would thus be limited to the same materials sampled in the same way.

Now coming to a discussion of *continuous mixing* first of all we must realize that there are some striking differences between this process and batch mixing. Therefore the method of studying continuous mixing must be different from an investigation of the batch process.

With continuous mixing we deal with a steady-state process, that is to say the degree of mixing at a certain point in the mixer does not change with time. However it must be taken into account that it may cost considerable time to arrive at such a steady-state, which makes the experiments the more difficult. Furthermore the process is influenced by the constancy of the feed streams; variations in these streams effect the process throughout the mixer. This phenomenon causes some difficulties with experimental studies too.

In any continuous mixer one can expect that besides transverse mixing a certain amount of longitudinal mixing takes place. The combined effect of these two processes can be studied by measuring the degree of mixing as a function of the length of the apparatus. The degree of longitudinal mixing taking place can be observed from the residence times distribution.

Up till now the process has been dealt with in a theoretical manner. As far as we know there are no published studies on experimental work, either on the degree of mixing as a function of the length, nor of the residence time distributions. These are therefore the main objects of our study.

There is still another specific problem with continuous mixing that makes such an investigation difficult. The content of a continuous mixer is relatively small, and of our experimental mixers very small; therefore one must use special sampling techniques and methods of treating the results. Such techniques and methods had to be developed.

Now we shall briefly review the literature on the theoretical aspects of continuous mixing.

When a material is allowed to flow continuously through a piece of processing equipment, the relative times taken by the different elements of material to pass through the processing vessel is rarely found to be constant. Some of the elements may remain in the vessel for extremely short periods of time or on the other hand the period of retention can become extremely long. Since such variability in holding or residence times definitely affects the technical behaviour of continuous processing equipment, knowledge about this variability is always of value for the purpose of design and analysis of the performance of such equipment.

The generally adopted procedure for acquiring such knowledge involves investigating the reaction of the system under consideration when either one of its input streams suffers a momentary disturbance, or when a property of the inflowing material undergoes a sudden change from one steady value to another; as for instance when the colour changes from red to blue. In the latter case, if the fraction of blue $F(t)$ in the outflow is determined at different times t, then a plot of $F(t)$ against t will result in some form of curve whose shape depends on the relative times taken by the various portions of the material to flow through the vessel. Such curves are referred to as residence time distribution curves.

The use of the residence time distribution for the characterization of continuous flow systems was introduced in its general form by DANCKWERTS (1953b). Further fields of application have been recently pointed out by DE BAUN and KATZ (1961) to be: in analysing the behaviour of reactor systems, in investigations of the strictly transient behaviour of mixing system, in working out the behaviour of various systems from the kinetic point of view and in the solution of design problems which involve the smoothing characteristics of mixing tanks. In as far as the purpose of the present work is concerned, interest

will be limited to the methods used for defining an observed or empirical residence time distribution.

One of the simple cases which rarely occurs in practice is that of piston flow. In this type of flow the elements of material are considered to enter the system at the same moment, flow together in parallel paths through the system and finally leave at the same moment. In this type of flow all of the material elements have the same residence time with zero variance. On the other hand the variance has a very high value for perfect mixing. Perfect mixing is considered to occur when the elements of material as they enter the vessel are completely mixed, so that the properties of the material in the vessel are uniform and identical with those of the outgoing stream. The residence time distribution for perfect mixing has been shown to have the following mathematical form:

$$F(t) = 1 - e^{-t/\bar{t}}$$

Here the time t is divided by the mean residence time $\bar{t}$, in order to make the form of the equation valid for all cases. The mean residence time is usually taken to be equal to the volume of the vessel occupied by the material divided by the volumetric flow rate or the mass of material in the vessel divided by the mass flow rate. In between these two extreme cases the residence time distributions can have an infinite number of forms due to longitudinal mixing. The degree of longitudinal mixing taking place in the system can be studied graphically by means of what DANCKWERTS (1953b) referred to as the F-diagrams. This is simply a plot of $F(t)$ against $t/\bar{t}$.

An interesting distribution of residence times is that obtained when material is allowed to flow through a cascade of perfect mixers. Such a distribution can be represented by an equation of the following form:

$$F(t) = 1 - e^{-nt/\bar{t}} \left(1 + \frac{nt}{\bar{t}} + \frac{1}{2!} \left(\frac{nt}{\bar{t}} \right)^2 + \ldots + \frac{1}{(n-1)!} \left(\frac{nt}{\bar{t}} \right)^{n-1} \right)$$

where $n =$ number of perfect mixers.*)

A more practical approach for defining such a distribution of residence times is that recently proposed by DE BAUN and KATZ (1961). Their method is based on approximating the residence time distributions in terms of the probability distributions of the χ^2 – statistics. The random variable χ^2_f (f degrees of freedom) is defined as the sum of the squares of f independent Gaussian random variables, each with zero mean and unit variance. The proposed approximation is made in the form of multiples of χ^2, in the form of $1/2 \; \theta \chi^2_{2n}$, which is interpreted as the residence time in a cascade of n stirred vessels, each of nominal residence time θ.

The random variable $1/2 \; \theta \chi^2_{2n}$ has the probability density:

$$K(t) = \frac{(t/\theta)^{n-1} \; exp \, (-t/\theta)}{(n-1)! \; \theta} \qquad t \leqslant O$$

*) Care should be taken not to confuse the n mentioned here with that which represents the number of particles per sample mentioned at different places of the text.

with mean and variance given by:

$$\text{mean} \int_0^\infty t \cdot K(t)\, dt = n\,\theta$$

$$\text{variance} = \int_0^\infty (t - n\,\theta)^2 \cdot K(t)\, dt = n\,\theta^2$$

According to DE BAUN and KATZ (1961), a systematic approximation scheme for application of the previous concepts is made by estimating the mean μ and variance σ^2 of the observed sample distribution by some appropriate numerical or graphical method, and then to take the n and θ of the probability density as:

$$\theta = \sigma^2/\mu$$

$$n = \mu^2/\sigma^2$$

A special case of continuous mixing which has been dealt with is that of a *continuous flow blender*. Perhaps the best way to show the line of thought followed in this direction is made by mentioning the description of a continuous flow blender as reported by DANCKWERTS (1953b). According to him, a continuous flow blender is a mixing vessel into which flows a stream of material of continuously varying composition. In the vessel elements of material which have entered at different times are mixed, so that the outflowing stream shows less variation in composition than the input.

One of the first authors to deal with continuous flow blenders as previously described was BEAUDRY (1948). He considered the special case of a perfectly – mixed blender with a feed consisting of batches of material of finite volume. DANCKWERTS and SELLERS (1951) and DANCKWERTS (1953b) extended the treatment to cover the more general case where the feed is not finite in volume. Other notable contributions were made by KATZ (1958) who gave an expression which allows the determination of the mean residence time necessary to lower the variance of the inlet stream to a desired value when the time lag necessary for the decay of dependence of the concentration at neighbouring points is known, and by DE BAUN and KATZ (1961) who gave similar expressions for the flow of material through a cascade of perfect mixers.

In general it can thus be said that the theoretical approach to continuous mixing has only been limited to the definition of the process as given above. The problem of the continuous mixing of two or more streams of constant composition to produce a complete mix does not seem to have been dealt with. VAN DE VUSSE (1953) gives a very elementary discussion of the continuous mixing of liquids.

In the previous pages an attempt has been made to review the general theoretical aspects of batch mixing and continuous flow systems. The review is not exhaustive but rather selective in character. Furthermore, experimental investigations have only been barely referred to, these when necessary will be dealt with in later sections. The object of the review was to investigate the different possibilities which can be applied to the continuous mixing of solids. The main conclusions that can be drawn can be summarized in the following:

1. Mixing of materials with different physical properties is accompanied by very complex phenomena that are extremely difficult to handle quantitatively

from both points of view of testing for mixture quality and carrying out rate studies.

2. Since the present study deals with the continuous mixing of solids about which not much is known, it would be inappropriate to complicate the study by introducing the complex phenomena of mixing materials with different physical properties. Instead a simple material should be used and interest should be mainly directed to machine characteristics rather than material properties and their effect on the mixing process. For simple materials with uniform particle sizes and possessing no segregating tendencies the most appropriate measure of mixture quality is the Chi Square statistic. Actually for the conditions under which the Chi Square can be applied, it represents the most reliable of the measures of mixture quality which can be used until now.

3. The degree of longitudinal mixing taking place in a continuous flow system can be investigated from the residence time distributions of the material. Not all types of these distributions can be accurately described mathematically. When the observed distributions are complex they can be studied graphically by means of F-diagrams. For the special case where the distributions can be described in terms of a cascade of perfect mixers, the most appropriate method available at the moment is that of DE BAUN and KATZ (1961), which is based on approximating the residence time distributions in terms of the probability distributions of the χ^2 statistics.

CHAPTER 3

EXPERIMENTS WITH A MIXER, EMPLOYING A MECHANICAL MIXING ELEMENT

3.1. INTRODUCTION

Of the two mixers to be investigated, the one employing a mechanical element to produce flow and mixing will be dealt with in this chapter. It is a pilot plant model of a mixer used in industry (manufactured by SPAANS-MACHINE-FABRIEK, Hoofddorp). As mentioned already in chapter 2 with a continuous mixer it is necessary to study the degree of mixing as a function of mixer length as well as the distributions of residence times. Also because the hold-up of a continuous mixer is relatively small, and of our experimental mixer very small, special sampling techniques will be used.

3.2. EQUIPMENT, MATERIAL AND METHODS

Description of the Mixer. The mixer used consists of a horizontal U-shaped trough in which is located a mechanical mixing element. This latter comprises an inner screw conveyor of small diameter around which a metal strip is wound in the form of a helical ribbon. The ribbon is welded to small cylindrical metal rods which in turn are welded to the screw at appropriate positions so that there is a distance between screw and ribbon. The ribbon is provided to improve the mixing by disturbing the smooth flow of material produced by the screw. The whole assembly is so constructed that the ribbon acts to obstruct the flow of a portion of the material transported by the screw conveyor.

The stainless steel U-shaped trough is 150 cm long, 15.2 cm wide and 16.5 cm deep at the center line. It has four discharge openings located in its bottom at distances of 57, 82, 107 and 132 cm from the drive end of the machine. Along the outer wall of one side of the trough, a metal strip graduated in centimeters is fixed in order to facilitate the determination of the distance at which sampling is performed. The walls of both ends of the trough are cut to a rectangular shape which can closely accommodate the sliding metal ends of the shaft of the mixing element. The openings are sufficiently deep to allow a small clearance to be made between the rotating ribbon and the bottom of the trough.

Two designs of stainless steel mixing elements differing in the measurements of the pitches and the diameter of the screw conveyor were used. The more closely pitched mixing element will be referred to by I, while the other will be indicated by II. The measurements of the different parts of the mixing elements are given in table 1, while in plate 1 the two types are compared. It should, however, be pointed out here that the diameter of the screw reported in the table for the mixing element I, only refers to the first 80 cm of its length, while for the rest it was 10.4 cm.

The reason for this difference was that the initial diameter of the screw conveyor could not handle the experimental material properly and flooding usually took place even at low feed rates. To correct this fault the helicoidal surface of the conveyor had to be increased by an extra strip of metal. This enlargement was not uniformly carried out, but this was of minor importance because in most of the trials carried out with mixing element I the second outlet of the mixer was used. Throughout most of this distance the diameter was more or less uniform at a value of 10.9 cm.

TABLE 1. Measurements of different parts of the mixing elements.

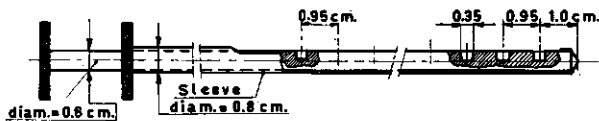| Mixing element | Screw shaft | | Screw conveyor diam. (cm) | Pitch length | | Width of ribbon (cm) | Outer diam. of ribbon (cm) | Clearance made with trough wall (cm) |
	Length (cm)	Diam. (cm)		Screw (cm)	Ribbon (cm)			
I	150	3.5	10.9	7.5	10	1	15	0.2
II	150	3.5	10.5	14.8	21	1	15	0.2

A variable speed motor directly coupled to the shaft of the mixing element was used as drive. Provision was made to allow easy disengagement. In this way the mixing element could be removed for sampling purposes and changed.

Feeding of the mixer was performed by means of two electromagnetic vibrators. The frequency of the vibrations could be adjusted to deliver the desired quantity of material. The vibrators were carried on a metal shelve on both sides of the U-shaped trough. Two metal funnel shaped hoppers fed the material directly to the vibrators. During the trials, the hoppers were kept completely and continuously full.
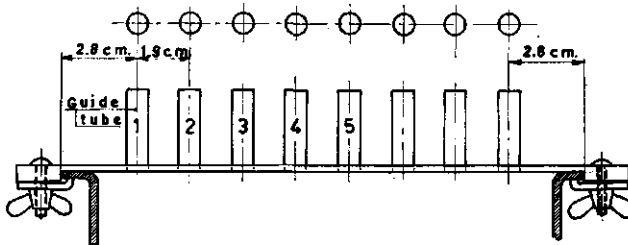
The whole mixer assembly was placed on a table of sufficient height to allow a weighing scale with container to be placed under the discharge outlet. A part of the mixer assembly is shown in plate 2.

Sampling Equipment and Procedure. The sampling equipment (see figure 1) comprised a sampling thief (1) and guide (2) and a sample collecting device (3). The thief consisted of a cylindrical rod 0.6 cm diameter with 15 cavities of
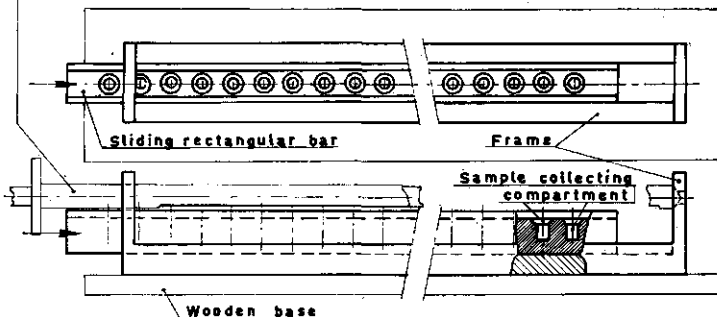
FIG. 1. Sampling equipment, the indicated numbers refer to the different parts described in the text.

0.35 cm diameter drilled at 0.95 cm intervals. Two rods were used, a brass one with cavities 0.2 cm deep and a steel one with 0.3 cm deep cavities. The first cavity was 1.0 cm from the end. The rods fitted into an outer sleeve, which had a strip 0.5 cm wide cut away along its length so that the cavities in the rods could be either covered or open.

The sampling guide had a brass base acting as a support for eight guide tubes, located at intervals of 1.9 cm. During sampling the guide could be firmly fixed to the upper open end of the mixer's trough by means of two clamps. It is shown in the sampling position in plate 2.

For sampling, the guide was fixed at the desired location. With the holes covered the thief was then carefully introduced into the mixture and when in position the samples were trapped and the thief was carefully withdrawn.

Particles trapped by any one cavity were collected without contamination by particles from an adjacent cavity by means of a specially constructed device. This consisted of a metal

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frame fixed to a wooden base, into which a rectangular brass bar could be closely fitted. The bar was bored into compartments each of which corresponded to a cavity of the sampling thief. The compartments could be closed by a sliding cover. Above the bar the frame was extended upwards at both ends and these were drilled with openings into which the sampling thief could be introduced in a horizontal position above the sample collecting bar.

To collect the samples, the bar and thief were introduced into the frame at their appropriate places. When in position the cavities of the thief were uncovered, whereupon the particles fell into the receiving compartments situated below. The thief and the bar were then removed from the frame and the compartments of the bar were closed by their cover. To remove the samples each compartment was uncovered in turn by sliding the cover backwards and the exposed sample was poured into an ordinary test tube of 1.8 cm diameter. All test-tubes were previously labelled to indicate number, distance and position of the sample which they would contain.

For counting the contents of each test-tube were evenly dispersed, by gentle tapping on the tube, along the surface of a white piece of paper. The number of particles of each colour was then counted by the aid of a magnifying glass and the results were registered in tabular form. From the counts the proportions of the components were determined and these were used for further treatment of the data.

Irrespective of the exact sampling pattern followed, the samples were either collected after careful removal of the mixing element or in the presence of the element.

Experimental Material. An experimental study of continuous solids mixing differs from that of a batch process in the fact that much more material is required. Thus, where a choice of an adequate material for batch studies would not result in practical difficulties, this is not the case for studies carried out on a continuous process.

Two materials were actually used, respectively ground glass and sand in different colours. The glass was used for the first trials only and was later abandoned because it could not be purchased clean and in one colour.

The glass was purchased in white and brown batches and was further treated in the laboratory in order to render it suitable for the purpose of the study. It was necessary to have it closely sized to an appropriate particle size, by means of a vibrating sieve. The size chosen was such that the particles were sufficiently small so that each sample contained an adequate number of them and at the same time were appropriately large to allow easy counting. It was decided that the best particle size for our purpose was that ranging between 0.4 and 0.6 mm in diameter.

Furthermore the obtained mixtures were reused after colouring with ink. For this purpose the glass was spread in shallow layers and the ink was applied by air spraying. The material was then allowed to dry overnight and on the following day it was sieved again in order to remove the agglomerated particles which were much coarser than required.

Treatment of the sand was much facilitated by the availability of a continuous vibrating sieve. This material was also closely sized to particles with diameters between 0.4 and 0.6 mm. The sand had first to be dried in a tray drier. Further treatment of the sand involved passing it several times over the continuous sieve, washing several times in running water, colouring by letting the sand stand for two days under an aqueous solution of the pigment and finally collecting the coloured sand on trays and drying. The sand was coloured red and blue by Waxoline Rhodamine (ICI) and Methylene Blue respectively.

Sand mixtures from the different trials were also used again. This was performed by washing in running water which removed the red but not the blue pigment. The sand thus treated was coloured again by Methylene Blue to provide new quantities of blue sand. To acquire red sand fresh batches had to be treated as previously described every time. Several physical properties of the glass and the sand are given in table 2.

TABLE 2. Physical Properties Of The Experimental Materials.

Experimental material	True density g/cm³	Bulk density g/cm³	Percentage voids	Angle of repose	Particle size (mm)
Glass	2.51	1.21	62	36°	0.4–0.6
Sand	2.64	1.42	46	34°	0.4–0.6

Adjustment of Feed Rate. When the vibrators were tested it was observed that a very wide range of feed rates could be produced. In most of the trials we preferred, however, to use low feed rates in order to reduce the quantity of experimental material to a minimum, since such material was difficult to acquire.

For adjustment, each vibrator was tested separately. To shorten the time required for the tests, the speed of the mixer was fixed at an high level and the feed hopper of the vibrator concerned was filled with material. The mixer and the vibrator were then allowed to operate and after some time, the time necessary for discharging 20 kg of material was determined. By repeating the procedure for each vibrator several times and making the necessary adjustments, it was possible to get a more or less constant feed rate.

When the feed rates were observed to be constant, the time required by each vibrator to deliver 10 kg and the time required by both vibrators to deliver 20 kg of material was determined before every experimental trial. Typical results of such a check are given below:

Vibrator	Output	Time	
		min.	sec.
I	10 kg	8	42
II	10 kg	8	32
I + II	20 kg	8	42

For the results given above the constant feed rate was considered to be equal to 2.3 kg/min. In a similar manner the other feed rates were determined.

3.3. INVESTIGATION OF THE DEGREE OF MIXING

In principle, the performance of the machine as a mixer of granular materials could be investigated by studying the quality of the mixture at the outlet or by studying this quality in the mixer itself. We decided to use the latter procedure only, because of the following reasons:

1. The analysis of the mixture while inside the mixer gives a good picture of the mixing pattern of the machine.
2. Such a procedure allows the determination (if any) of the relation between distance and mixture quality.
3. Sampling of the outflow is difficult to perform without disturbing the quality of the mixture.
4. The quality of the mixture near the outlet must reflect the quality of the mixture in the outflow and there is no need to sample the latter.

In mixing studies the most suitable sampling procedure is best determined experimentally. It is not possible to decide beforehand about such things as a good sampling plan or the number of samples which would give satisfactory results. It is true that the number of samples required for a certain degree of accuracy can be calculated by making use of statistical concepts, but the figures thus derived have little value because they are usually prohibitively high, at least as long as there is no mechanical device to do the analysis*. Furthermore, one is faced with the problem of adapting the sampling procedure to the general constructional features of the machine studied. It may also be that one only requires to inspect certain places of the machine which are suspected not to function properly. For these reasons and because nothing was known about

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*) It was tried to develop a mechanical device for counting particles of different colours but without success.

TABLE 3. Data on test trials

Trial number	Speed (r.p.m.)	Feed rate (kg/min)	Overall proportion determined by sampling entire material fed to mixer		Number of strips used and the							
			Proportion coloured glass (P)	Number of samples used	Total				Distan			
I	30	2.3	0.486	94	10	3	16	29	42	55		
II	60	2.3	0.497	96	17	5	11.5	18	24.5	31		
III	30	2.3	0.522	166	10	3	13	23	33	43		
IV	12	1.6	0.302	103	16	3	5.5	8	10.5	13		
V	10	1.7	0.285	132	19	3	5.5	8	10.5	13		

Remarks: sampling performed after removal of mixing element – places of feeding and outlet of mixer are schemati

the operating characteristics of the investigated mixer, it was decided to start the study with several test trials.

Test Trials. The results of five trials performed for this purpose will be discussed. For all five trials, mixing element I and glass were used.

Prior to every trial the feed rate was adjusted to the desired level by the procedure described in 3.2; the speed at which the mixing element was to be operated during the trial was also chosen.

Sampling was performed on the mixture as it lay in the mixer's trough and in such a way that it conformed with the general features of the accumulated layer. In order to clarify this point it is perhaps convenient to describe the manner by which the material tended to accumulate in the mixer.

From the moment that material is fed into the mixer, the feed streams tend to accumulate at the place of their entry. At a certain feed rate and speed of the mixing element, the material gradually accumulates to a layer whose depth is ultimately determined by the opposing actions of the inner screw and the outer ribbon. When the sizes of the slugs transported forth and back are equal, no net transport in the forward direction takes place. However, as more and more material enters the mixer, the quantity of material conveyed by the screw increases and surpasses that which can be handled by the ribbon, thus resulting in a net transport in the forward direction. A steady-state is established when the excess transport capacity of the screw is equal to the quantity of material fed to the mixer. If the rate at which material is fed to the mixer exceeds that which can be handled by the mixing element flooding takes place. The transport capacity of the screw conveyor could be greatly increased by increasing the rotational speed, so that the mixer used could actually handle very large feed rates.

By the effect of the rotating screw, the layer of material in the trough tends to collect on one side of the axis of the mixing element and assumes an inclined position.

Furthermore, the layer acquires a wavy appearance due to the action of the ribbon and screw. Less of the material is present at those places where the spiral and screw remove their slugs, while more of the material is present at those

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distances from the feed end													Sampling pattern in the strips						Mean no. of particles per sample (n), as determined from all samples analysed
from feed end in centimeters													Total no. of samples from five points	No. of samples per point					
1	94	107	120											10	2	2	3	2	1	–
4	50.5	57	63.5	70	76.5	83	89.5	96	102.5	109				15	2	4	4	3	2	–
3	77	97	117											10	2	2	3	2	1	88
8	20.5	23	25.5	28	30.5	33	35.5	45	60					20	3	5	5	5	2	130
8	20.5	23	23.5	28	30.5	33	35.5	38	40.5	43	45.5	60		20	3	5	5	5	2	136

strated on figures expressing results.

places where the slugs are delivered. This phenomenon can be clearly seen from plate 2.

For sampling, the mixing element was carefully removed from the trough. The mixture was sampled in transverse strips at definite intervals throughout the length of the mixer. Because of the manner in which the material tended to arrange itself in the mixer, only five positions of the sampling guide could be used. The number of samples removed from every position depended on the depth of the material as well as on the total number of samples that were required from every sampling strip. Information on all trials is summarized in table 3.

As the streams of materials delivered by the vibrators could not be counted upon to give an accurate estimate of the proportions of the different glasses mixed, it was decided to acquire such information by sampling the entire batch of material fed to the mixer during the trial. For this purpose the already sampled mixture lying in the mixer's trough was added to the mixture discharged and both were well mixed. Small batches from the resulting mixture were then removed and individually sampled. The number of samples collected and analysed for this purpose for every trial are given in table 3. The mean of these sample proportions was taken as an estimate for the overall proportion of coloured glass.

The first test trial was performed with a speed of 30 r.p.m. and a feed rate of 2.3 kg/min. The vibrators were adjusted to deliver brown and white glass in a proportion close to 1 : 1. Actual sampling revealed that the proportion of brown to white glass was 0.486 : 0.514.

A second trial was carried out with 60 r.p.m. Although the feed rate was again 2.3 kg/min, the proportion of brown to white was now found to be 0.497 : 0.503.

Thus for the same adjusted positions of the feed vibrators, the proportion of brown to white glass was not constant but tended to vary from trial to trial. The difference was, however, not excessive and could be ignored for all practical purposes.

In these two trials, the vibrators were placed on opposite sides of the mixer's

trough at about 15 cm from the feed end. They were placed in such a position that their discharge ends did not extend beyond the inner wall of the mixer's trough. By such an arrangement it was hoped that mixing of the entering streams could kept at a minimum. For both trials the last outlet opening was used.

The mixtures of the two trials were sampled differently. In the first trial ten samples were taken from each of ten sampling strips. Sampling was started at 3 cm from the feed end, and was repeated at intervals of 13 cm up to a distance of 120 cm. In the second trial the sampling strips as well as the number of samples per strip were increased. Sampling was started at 5 cm and was continued to 109 cm from the feed end of the mixer; in total 17 strips were sampled. The number of samples in every sampling location was increased to 15.

The proportion of brown glass in the samples was determined from counts, and the mean of these sample proportions was then computed for every sampling strip in order to compare the values with the overall proportion determined from sampling the entire batch of material fed to the mixer during the trial. The results for the two trials are reported in figure 2, in which the mean proportion brown glass is plotted against the distance at which sampling was performed.
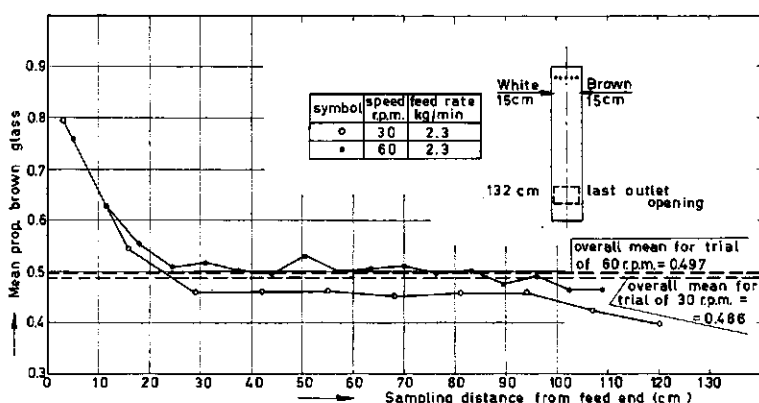

FIG. 2. Test trials I & II. Mean proportion of brown glass as function of distance at which sampling took place.

This figure shows that the local means follow the same pattern in the two trials. At the early part of the mixer the mean proportion of brown glass is high, while in the last part of the mixer it is low. In between, the local means tend to fluctuate about a more or less constant value, which is near to the overall proportion for the trial with a speed of 60 r.p.m., but which strongly differs from the overall proportion for the trial with a speed of 30 r.p.m.

The excess of brown glass in the early part of the mixer may be explained from the fact that the mixing element in this part is asymmetrical with respect to the entering feed streams. Thus, whereas the screw conveyor tends to transport more white than brown in a forward direction, the ribbon on the other hand, causes much more brown than white glass to accumulate in this part of the mixer. Under these conditions the quantity of brown glass in the other parts of

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the mixer should be less than expected. By reference to figure 2 it will be observed that this is indeed the case for the trial with a speed of 30 r.p.m.

It is also evident from this figure that the material nearest to the mixer's outlet had the least amount of brown glass. This indicates that insufficient time had been allowed for the mixer to discharge the first quantities of material fed to it, since these quantities contain the least amount of brown glass, because of the accumulation that took place at the beginning of the mixer.

From these considerations it follows that the mixer was not yet operating at a steady-state. The better agreement between the local proportions and the overall proportion for 60 r.p.m. than for 30 r.p.m., indicates that a steady-state can be reached much quicker at a speed of 60 r.p.m. than at a speed of 30 r.p.m.

There is no doubt that if the mixer was allowed to operate for a sufficiently long period of time, a more favourable distribution of the two colours of glass could be acquired. However, such a procedure was considered to be inadequate, because it would require large quantities of experimental material. Instead it was decided to change the place of the feed vibrators to another place which allowed less of the brown glass to accumulate in the early part of the trough.

Accordingly, the place of the vibrators was moved back from 15 cm to about 3 cm from the feed end of the mixer's trough. In order to test the effect of this change a trial III was carried out with a speed of 30 r.p.m. and a feed rate of 2.3 kg/min. Information concerning this trial is given in table 3 and the results are reported in figure 3.

In the lower part of this figure the local mean proportions of brown glass are plotted against distance. From the graph it can be seen that the change in feed place succeeded to bring about some improvement in the results. Less
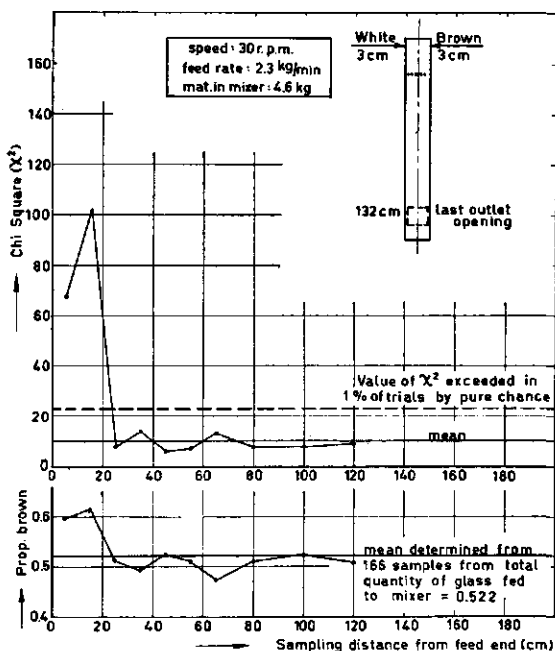

FIG. 3.
Test trial III. Lower graph, plot of composition vs. distance. Upper graph, plot of Chi Square (χ^2) vs. distance.

brown is accumulated at the feed end of the trough and the downward trend in composition observed near the outlet in the previous trials is absent. Throughout the greatest part of the mixture the local mean proportions of brown glass, although still on the low side, did not widely differ from the overall proportion of brown glass. To test whether the sample proportions at the various sampling places were significantly different from the overall proportion, Chi Square (χ^2) values were computed from the ten sample proportions of every sampling strip. The form of the Chi Square used was as follows:

$$\chi_N^2 = \frac{n \sum\limits_{i=1}^{N} (x_i - P)^2}{P(1 - P)}$$

where P = overall mean proportion of brown glass as determined from sampling the entire quantity of material fed to the mixer during the trial.

n = overall mean number of particles per sample as determined by averaging the number of particles per sample for all samples removed during sampling of the mixture.

x_i = observed proportion brown glass in the ith sample.

N = degrees of freedom, equal to number of samples used.

The calculated values of Chi Square are plotted in the upper part of figure 3. The line called mean represents the expectation of Chi Square which is equal to the number of degrees of freedom used. This is $N = 10$. The proportion of brown glass is estimated by the overall proportion as determined by sampling. The broken line represents that value of Chi Square with ten degrees of freedom which is exceeded by pure chance in one percent of the cases (i.e. based on a one tailed test for a 99 % confidence level).
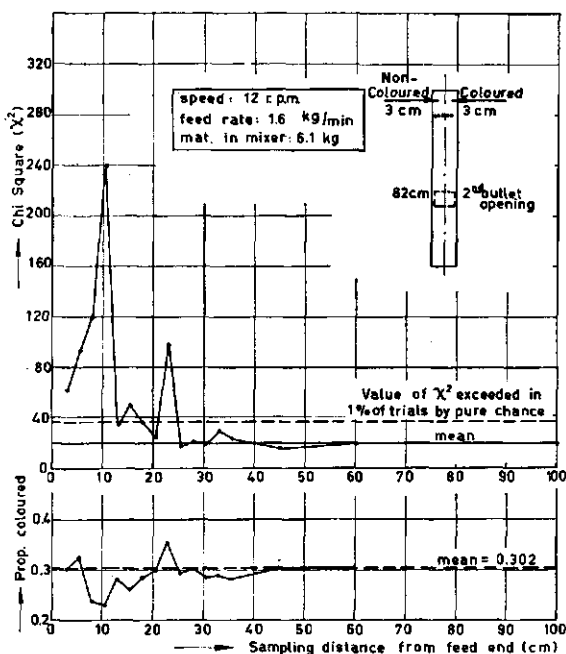
By reference to figure 3 it will be observed that only the first two sampling distances had sample proportions which were significantly different from the overall proportion of 0.522. However, the difference is not due to a non-random distribution of equal numbers of brown and white particles but it is due to the presence of brown glass much in excess of the mean value expected for any part of the mixer.

To test whether the undesirable accumulation of one component could be decreased still more, two other feed places were tried. Also because the mixing now seemed to be complete at short distances from the feed end for speeds of 30 r.p.m. and possibly higher, it was decided to use lower speeds.

In both trials IV and V coloured and non-coloured glass was used. Furthermore to use less of the coloured glass, the feed rate of the vibrator delivering this material was decreased and the second outlet of the mixer was used instead of the last.

In trial IV, which involved a speed of 12 r.p.m. and a feed rate of 1.6 kg/min, the vibrator troughs were made to deliver their feed streams very near to the axis of the mixing element at about 3 cm from the feed end. The mixture of this trial was sampled at intervals of 2.5 cm up to a distance of 35.5 cm and then only two more places were sampled at distances of 45 cm and 60 cm respectively. The number of samples in every sampling strip was increased to twenty because of the greater quantity of material which accumulated in the mixer than in previous trials. The results are reported in figure 4.

FIG. 4.
Test trial IV. Lower graph, plot of composition v.s. distance. Upper graph, plot of Chi Square (χ^2) v.s. distance.

From the figure it can be seen that the local means (and accordingly the Chi Square values) tend to fluctuate between high and low values and are only in good agreement with the overall proportion at a few places. A notable difference between the results of this trial and those of the previous one is that the first two sampling strips showed mean values which do not differ much from the overall mean. The erratic behaviour of the local means at other places of the mixture indicates, however, that the new feed place was far from satisfactory.

In the final test trial V, one of the vibrators was extended in length so that it delivered its stream close to that of the other vibrator at the righthand corner of the mixer's trough. As with the previous trials, the place at which the mixer is fed, is schematically illustrated at the top righthand corner of figure 5, and the information concerning this trial is reported in table 3.

From the lower graph of the figure it can be seen that the overall proportion is in good agreement with the local means, although it is still slightly higher than most of them. This is especially true for the first sampling place, where it can be observed that the coloured glass is obviously lower than the average. On the other hand, the excessive fluctuations in the Chi Square values, as seen in the upper graph of the figure, indicate that the sample proportions at any one place can differ widely from the overall proportion. It is difficult to explain why the mixing is good in some places and bad in others. A very probable explanation is that mixing with this low r.p.m. is actually bad throughout the mixer, but during the removal of the mixing element for sampling purposes some places are improved by the disturbance caused by the different parts of the mixing element as they leave the mixture.

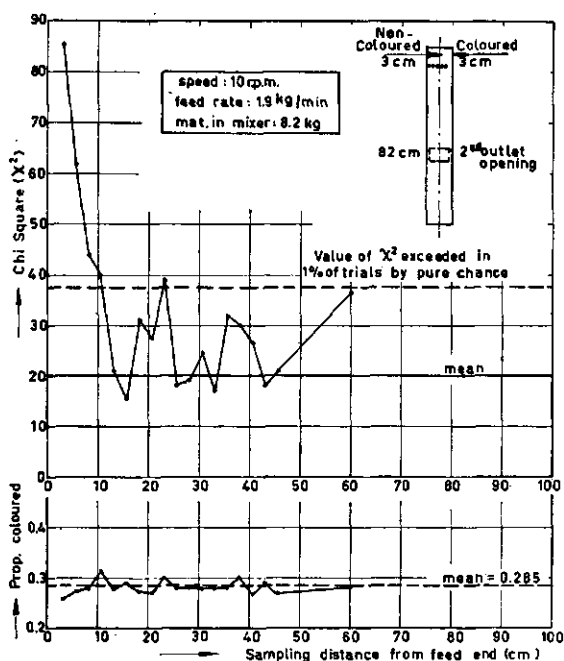Taking the results of these test trials together we draw the following conclusions:

FIG. 5.
Test trial V. Lower graph, plot of composition vs. distance. Upper graph, plot of Chi Square (χ^2) vs. distance.

1. The first part of the mixer can be considered for all practical purposes as a "dead space". It can be reduced to a minimum by choosing an appropriate feeding place.

2. This "dead space" affects the performance of the mixer only during the early period of operation after the start of the machine. However, once filled with material this "dead space" will have no further effect.

3. The mixture must only be sampled after steady-state operation has been established. By steady-state is not only meant equality of the inflow and out-flow streams, but also the absence of the residuals of disturbances in the composition of the flowing stream such as that caused by the "dead space".

4. Sampling must be performed without removing the mixing element in order to avoid the excessive consequent disturbance.

5. The place of sampling has a great influence on the obtained information concerning the quality of the mixture.

6. Determining the overall proportion by sampling the entire quantity of material fed to the mixer during the trial, does not contribute much to the accuracy of the results. Some other less laborious procedure would most probably give an equally satisfactory estimate.

7. The influence of the speed, which is apparently the most important single variable affecting the performance of the machine, cannot be investigated by the procedure adopted so far for the test trials.

Mixer performance at different speeds. From the results of the previous trials no conclusion could be drawn concerning the effect of speed on the mixing process. For most of the speeds used, mixing seemed to be complete at distances of less than 30 cm.

Furthermore it has become obvious that in order to test for the true effect of speed, sampling should be performed without removing the mixing element and only after sufficient time has been allowed for the mixer to operate at a steady-state. For the purpose of checking on the steady-state the following procedure was adopted. During every trial the rate of outflow was determined several times. When the measurements indicated that no more accumulation was taking place, the mixer was assumed to be operating at a steady-state with respect to the inflow and outflow streams. To be sure of a true steady-state operation before sampling the machine was then allowed to run for some additional time.

Sampling without removal of the mixing element greatly reduces the space available for sampling, because the sampler could only be put into those places of the mixture, which were not shielded by the mixing element. To get a number of independent samples for estimating the local variability it was decided to collect the samples from replicates. That is, the mixer is operated until a steady-state is established, whereupon it is stopped and a series of samples is removed. It is then operated again and the same procedure as before is repeated to get a second series of samples. Of course this could be done several times, but actually each trial was replicated only twice.

In every replicate eight equidistant strips were sampled along the length of the mixer. At every sampling location the samples were removed from two sampling points approximately two centimeters apart. To distinguish between these two points, they will be referred to as the inner and the outer sampling points. This indicates their relative positions with respect to the axis of the mixing element. Both lie on the left side of the axis, occupying positions 2 and 3 of the sampling guide. Four samples were taken from every sampling point, and thus in total sixteen samples became available for every distance from the point of feeding. The composition of the samples was determined as usual from counts and the results were tabulated according to place and order of removal of the samples from the mixer. The overall mean proportion of blue sand was not determined by sampling the entire quantity of material fed to the mixer, but from those sampling strips which showed a more or less constant composition in every trial. This usually involved the last sampling strips of the trial, the exact number of which for every case can be found in table 4.

With this series of trials the vibrators were adjusted to deliver their streams close to each other at the left-hand corner of the feed end of the mixer at approximately 3 cm from that end of the mixer's trough. Sand coloured in blue and red formed the experimental material and the second outlet of the mixer was used in all trials. The trials were carried out with mixing element I.

In total five trials with different speeds of the mixing element were carried out. The experimental procedure adopted for all trials followed similar lines, apart from the minor specific differences which have been summarized in table 4.

The results of the trial with a speed of 9 r.p.m. are reported in figure 6. The lower part of the figure represents a plot of mean proportion blue sand against distance at which sampling was performed. At every sampling distance four mean proportions are plotted. These represent the average values of the four samples analysed for each of the inner and outer sampling points in the two replicates. The solid symbols refer to the outer sampling point, while the hollow symbols refer to the inner one. The broken line represents the mean proportion as determined from the last three sampling strips.

TABLE 4. Data on trials carried out to study effect of speed on mixer performance

Trial number	Speed (r.p.m.)	Feed rate (kg/min)	Overall Proportion	
			Proportion blue sand, P.	Determined from
I	9	2.5	0.503	last three strips
II	15	2.5	0.492	last three strips
III	23	2.5	0.488	last four strips
IV	40	2.5	0.511	last five strips
V	60	2.5	0.505	last seven strips

Remarks: sampling performed in presence of mixing element-mixer fed at same place for all trials as

In the upper part of the figure the magnitude of the variability in sample proportions at the different sampling locations is represented by a plot of Chi Square against distance. The mean value of Chi Square was calculated for 16 degrees of freedom and is represented by the solid line of the graph. The
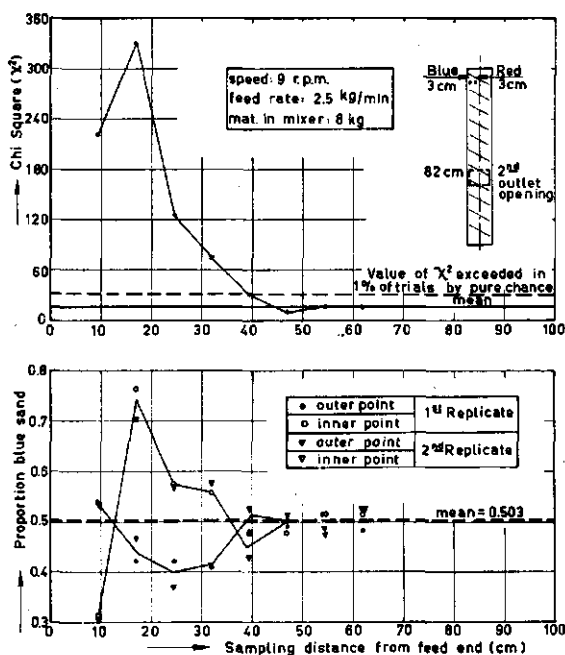

FIG. 6.
Mixing at low speed of 9 r.p.m. Lower graph, plot of mean proportion blue sand vs. distance for the two sampling points in the two replicates. Upper graph, plot of Chi Square (χ^2) vs. distance.

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al	No. of strips used and their distances from feed end								Mean no. of particles per sample (n) as determined from all samples analysed
	Distance from feed end in centimeters								
	9.5	17	24.5	32	39.5	47	54.5	60	158
	,,	,,	,,	,,	,,	,,	,,	,,	158
	,,	,,	,,	,,	,,	,,	,,	,,	149
	,,	,,	,,	,,	,,	,,	,,	,,	158
	,,	,,	,,	,,	,,	,,	,,	,,	155

ι the figures.

broken line represents the Chi Square value with 16 degrees of freedom which is exceeded in only one per cent of the trials by pure chance.

The lower graph of figure 6 shows that the mean proportions of the different sampling points are not the same. In the early part of the mixer the difference in composition is not only large but tends to follow a certain definite pattern as indicated by the lines drawn through the points. The mean compositions of the inner and outer sampling points strongly differ. Furthermore, the same trend is more or less reproducible in the replicates. With increasing distance, the compositions of the two points gradually approach each other until in the last three sampling strips the differences show no trend. Random fluctuations around a mean value will always be present because of the random sampling error. In the early part of the mixer the differences from the mean value are not random but in the last three sampling strips the fluctuations are random in character. These facts are clearly indicated by the Chi Squares computed for the different sampling strips and reported in the upper part of the figure.

Although the results of the other trials could be reported in a similar manner as that used for 9 r.p.m., this was not done because of the trend observed at a speed of 9 r.p.m. was limited to the first two sampling distances with 15 r.p.m. and did not occur with higher revolutions. A plot of Chi Square against distance was therefore considered to be sufficient to show the main differences between the various trials.

In figure 6 the Chi Square values for all the trials have been collected together to allow easy comparison. From the figure it can be seen that the estimated Chi Squares for the first sampling strips are high and then decrease with distance along the length of the mixer. The values of Chi Square at the same sampling strip are not the same for the different speeds. For any place in the mixture in which mixing is not yet complete, the Chi Square values tend to be lower for higher speeds than for lower speeds. Thus for the same place
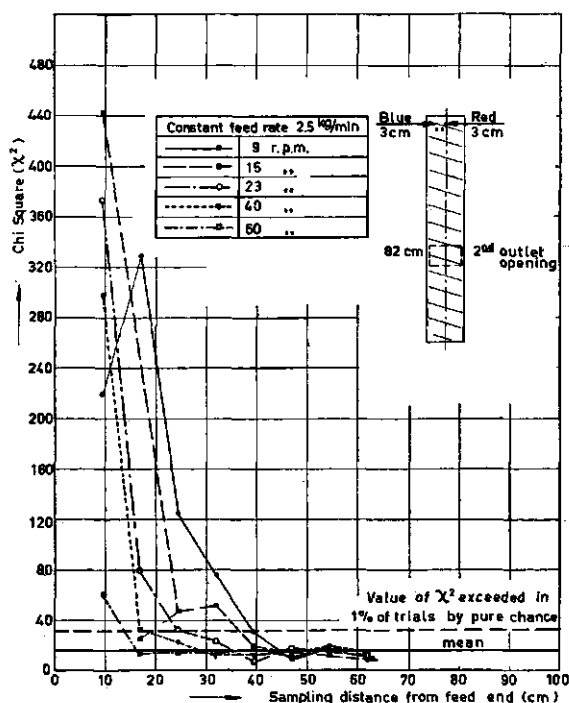
FIG. 7.
Chi Square (χ^2) vs. distance. Mixing element rotated at indicated speeds.

where mixing is not complete the mixture is relatively better mixed at high than at low speeds.

The reduction in Chi Square values between strips for the same speed or for the same strip at different speeds is very rapid. At high speeds the Chi Square values are almost instantaneously reduced to their mean value between the first and second sampling points, while at low speeds they are still sharply reduced to values not far from the mean in the very early part of the mixer.

For 9 r.p.m. the mean value of Chi Square is reached at the sixth sampling strip, while for 15 r.p.m. and 23 r.p.m. it is reached at the fifth sampling strip. The number of the sampling strip at which the mean value of Chi Square is reached is further reduced to the fourth for 40 r.p.m. and to the second for 60 r.p.m. Thus low speeds require longer distances than high speeds in order to bring mixing to completion. A speed of 9 r.p.m. requires about 30 cm more of mixer length than a speed of 60 r.p.m. For a feed rate of 2.5 kg/min and a speed of 9 r.p.m. mixing is still complete at a distance of less than 50 cm.

An interesting observation which can be seen from figure 7 is that a statistically non-significant Chi Square value for one point, does not mean that mixing is complete. Values of Chi Square which ceased to be statistically significant still continue to decrease with mixing until they reach the mean value after which they tend to show no further decrease. Taking some of these earlier points together yields of course again a significant difference from the mean.

When the previous results are considered together it is possible to draw the following conclusions:

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1. At very low speeds of the mixing element the material flows for relatively long distances without undergoing appreciable mixing.

2. At higher speeds a shorter length suffices for complete mixing. However, because the improvement in mixture quality in our experimental mixer is very rapid, it is impossible to define a relation of general interest between mixture quality (as expressed by a mixing index) and mixer length in a mathematical form.

Performance of the Widely Pitched Mixing Element II. Only one trial was carried out, at a speed of 23 r.p.m., a feed rate of 2.6 kg/min; the last outlet of the mixer was used. Sampling of the mixture was made from single points, at intervals of about 7 cm, starting at 5 cm and ending at 116 cm from the feed end. In one or two cases the sampling interval was slightly larger because the mixing element prevented introduction of the thief at the desired point. In total 16 points were sampled. The place of sampling was near to the axis of the mixing element corresponding with what has been referred to in the previous trials as the inner sampling point.

From each sampling point only three samples were collected. This was done by discarding the deepest sample removed by the sampling thief and then taking the following three samples. The sample proportions determined from the different sampling points are reported in the lower graph of figure 8. Each of the plotted points is indicated by a number which refers to the order of removal of the sample from the mixture, with number one indicating the deepest sample and the other two numbers refer to higher positions in a vertical direction.

The overall proportion of blue sand was determined by averaging all the sample proportions except the first three. It is represented in the lower graph
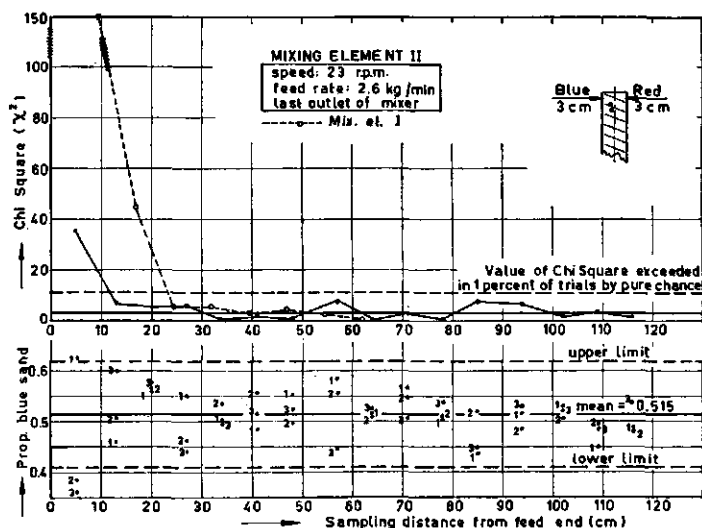

FIG. 8. Comparison between two mixing elements. Lower graph, plot of observed proportions of blue sand vs. distance for trial with widely pitched mixing element. Upper graph, plot of Chi Square (χ^2) vs. distance for the two mixing elements as indicated.

of figure 8 by the solid line, from which it can be seen that the overall proportion determined in this way represents the sample proportions in all places fairly well. The broken lines in the graph are the upper and lower limits of a 99 % confidence interval for sample proportions based on a Student-t value with 44 degrees of freedom. The mean number of particles used for computing the standard error of sample proportions was 160 particles as determined by averaging all the samples analysed.

From figure 8 it can be seen that only the sample proportions of the first sampling point fall outside the limits of the confidence interval. The Chi Square values represented by a solid line also lead to the same conclusion. Thus for mixing element type II, mixing seems to be complete at distances of less than 10 cm, for a feed rate of 2.6 kg/min and a speed of 23 r.p.m.

In order to compare the performance of the two mixing elements used in this study, the previous results with mixing element type I for a speed of 23 r.p.m. will be used. Since, however, only three samples were taken for type II, the same will be done for type I. For this purpose from the four samples of the inner sampling point only the last three will be used. Using all these sample proportions but the six of the first two sampling points, the overall proportion was determined to be 0.483, which value agrees well with the value of 0.487 found by averaging the results of 16 samples per sampling strip. In the same manner the mean number of particles per sample was found to be 152 which is again in good agreement with the value of 149 determined from many more samples. From these values the Chi Squares for the different sampling points were computed and are reported in the upper graph of figure 8 as represented by the broken line.

When the two plots of Chi Square are compared, it can be seen that the obvious differences between the two types of mixing elements are limited to the first part of the mixer. For the widely pitched mixing element the sample proportions are not significantly different after the first sampling point while for the second type of mixing element the differences are significant for the first two points. Thus type II seems to perform better than type I in this first part of the mixer. This conclusion is also in agreement with the observation, that in contrast to type I, the mixing element of type II allowed both colours of sand to accumulate in equal quantities in this part of the mixer.

This difference in performance is definitely related to the difference in the length of the pitches of the two mixing elements. For, whereas a widely pitched element will not interfere much with the entering feed streams, the same is not the case for a closely pitched element which tends to distribute the feed streams at the place of their entry in a more irregular manner. In the latter case it becomes more likely to find regions in this early part of the mixer which contain more of one of the sands being mixed. Apart from this obvious difference between the two mixing elements no further differences in performance could be observed. This should not be taken to indicate, however, that type II performs better than type I in all respects because of the following reasons. In the first place the results are based on the analysis of only three samples and this number is insufficient to provide accurate information about the degree of mixing. For example by reference to the upper graph of figure 8 it will be observed that in the case of mixing element I, using three samples instead of sixteen has accidentily resulted in a lowering of the Chi Square values of the third and fourth sampling distances, thus suggesting better mixing than is actually the case.

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This can be observed by comparing the Chi Square values reported for the same sampling distances in figure 7. In the second place the material used for the experiments has very good flow properties and is thus very easily mixed. If a less easily mixed material is used it may happen that the two mixing elements would then perform differently. Keeping in mind these facts we may conclude by saying that after a distance of about 40 cm both mixing elements give their final mixture, and so they are equally good from this distance on.

3.4. RESIDENCE TIME DISTRIBUTION STUDIES

The very casual treatment which continuous solids mixing received in the literature seems to be based on the theoretical and experimental knowledge acquired from residence time distribution studies carried out in the field of fluids. For example LACEY (1954) stated that the concepts developed for batch mixing can be extended to the case of continuous solids mixing by the methods suggested by DANCKWERTS (1953b) for the characterization of fluid flow systems. RUMPF and MUELLER (1962) classified the methods used for mixing powders and they are of the opinion that the mixing efficiency of a continuous process is determined by such factors as transverse mixing, longitudinal mixing and the degree of fluctuations in the feed streams.

For continuous solids mixing it is clear, however, that the extent and the conditions under which these various postulations are valid is not known, and not well understood. Because of these reasons it was felt that some investigations in this direction would be worth while. In the coming sections we shall discuss the work carried out for this purpose.

Experimental Procedure: The procedure followed for determining the residence time distribution under different operating conditions of the mixer involved changing a steady stream of red sand to a stream of blue sand, and then determining the composition of the outflow at different times. For this purpose the feed rates of the two vibrators were very carefully adjusted to an equal and constant level. During the trial, first the stream of red was put on and after some time measurements on the feed rate were carried out at short intervals in order to check on the constancy of the outflow. When this was observed to be constant, the stream of red sand was changed to a stream of blue sand. The change-over of the streams was performed by means of two electric switches located side by side in a wooden box, and was more or less instantaneous. The moment at which the blue particles were put into the stream, was taken as the zero time of the measurements.

With the exception of one trial (involving a speed of 9 r.p.m.), the outflow was sampled at quarter minute intervals. This was done by moving a small metal container through the outflowing stream and then quickly and carefully pouring the removed samples into separate glass bottles. This was repeated until it was considered that sufficient samples were available to provide the necessary information.

When sampling was over, the vibrators and the mixer were stopped at the same time by means of a common electrical switch. The mixer was then emptied and the weight of the material present in it was determined.

Because of the relatively large size of the samples collected from the outflow at different times, they were again sub-sampled by the equipment described

in 3.2. The proportion of blue sand in the outflow at a certain time was determined by averaging the proportions of these sub-samples.

Scope of the investigations and method of treatment of the results. The effects of speed and length of the mixer on the residence time of the material were investigated. The residence time distributions for four different speeds were determined, using mixing element type I and the second outlet of the mixer. The proportion of blue sand was determined from five sub-samples taken from the initial samples collected from the outflow at different times. The four speeds investigated were 9 r.p.m., 23 r.p.m., 40 r.p.m. and 60 r.p.m. respectively.

In the second series of trials, different outlets of the mixer were used, viz. the first, second and last, while the speed was kept constant at 23 r.p.m. The proportions of blue sand at different times were determined from ten sub-samples and mixing element type II was used.

The results of all trials indicated that an appreciable amount of longitudinal mixing takes place. It proved that in most cases the observed distributions were analogous to the case of a cascade of perfect mixers. The most appropriate method of representation available for this case at present is that proposed by DE BAUN and KATZ (1961) which involves approximating an empirical residence time distribution by a Chi Square distribution. The principles involved have already been reviewed in chapter 2 and we shall limit ourselves to a description of the procedure followed in adopting this method to the treatment of our results.

For this purpose it is necessary to compute the mean (μ) and variance (σ^2) of the observed residence time distributions. This was done in the following way. The experimental points were plotted on logarithmic probability paper and the points were fitted by a smooth curve. From the fitted curve the cumulative percentage $F(t)_i$ corresponding to a set of times (t_i) were determined. Actually 19 pairs of values were determined in this way for every trial. From these values the mean (μ) and variance (σ^2) were then computed from the following approximate expressions:

$$\mu = \sum_{i=1}^{k} t_i \cdot \varphi_i \quad \text{and} \quad \sigma^2 = \sum_{i=1}^{k} (t_i - \mu)^2 \cdot \varphi_i$$
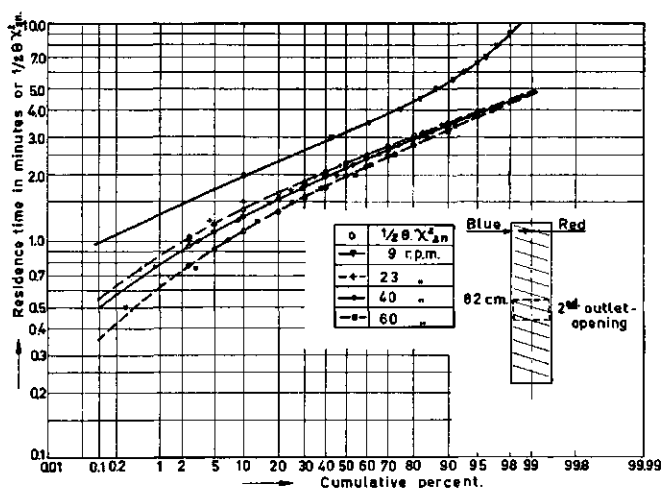
In these expressions the different φ_i's are defined by:

$$\varphi_i = \begin{cases} \frac{1}{2} [F(t)_1 + F(t)_2] & \text{for } i = 1 \\ \frac{1}{2} [F(t)_{i+1} - F(t)_{i-1}] & \text{for } i = 2, 3, \ldots, (k-1). \\ 1 - \frac{1}{2} [F(t)_{k-1} + F(t)_k] & \text{for } i = k \end{cases}$$

The values of μ and σ^2 were then used to compute the nominal residence time per perfect mixer $\theta \; (= \sigma^2/\mu)$ and the number of perfect mixers $n \; (= \mu^2/\sigma^2)$ corresponding to the observed residence time distribution.

In order to check to what extent these values agreed with the experimental results a plot of $\frac{1}{2} \theta \chi^2_{2n}$ against the cumulative percent corresponding to the different values of Chi Square with $2n$ degrees of freedom was made on the same graph of the experimental points. The cumulative per cent and their corresponding values of Chi Square were taken from the tabulated values reported by HALD (1960). The values given in this table are only for whole

FIG. 9.
Residence time distributions for different rotational speeds of the mixing element and their corresponding $\frac{1}{2}\theta\chi^2_{2n}$ approximations plotted on logarithmic probability paper.

numbers of degrees of freedom. However, when the analysis of the data resulted in a number of degrees of freedom which was not whole, the Chi Square values for such cases were either determined by interpolation between two whole numbers of degrees of freedom or when the difference from a whole number was not large the Chi Square values corresponding to the nearest whole number of degrees of freedom were used.

Discussion of the Results. By the methods discussed above the results of the seven trials for the different speeds and for the different outlets of the mixer were analysed. The data of the experiments and the results of the analysis are given in tables 5 and 6. The experimental points and the points determined by the Chi Square approximation are represented graphically in figures 9 and 10.
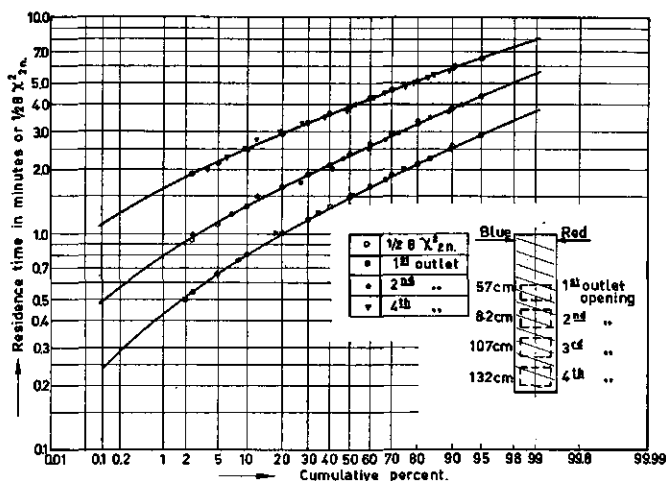

FIG. 10.
Residence time distributions for different mixer lengths and their corresponding $\frac{1}{2}\theta\chi^2_{2n}$ approximations plotted on logarithmic probability paper.

By reference to the figures, it can be seen that for the experiments with 23 and higher r.p.m. the observed residence time distributions can be fairly well represented by the Chi Square approximation to the distribution of a cascade of perfect mixers. The shape of the curve for 9 r.p.m. proves that this case is not analogous to a cascade of perfect mixers.

TABLE 5. Data for residence time distribution trials at different speeds and same mixer outlet.

Rotational speed, (r.p.m.)	Feed rate, W(kg/min)	Material in mixer, G (kg)	Nominal residence time, G/W (min)	Mean residence time of observed distribution, μ (min)	Variance of observed distribution, σ^2	Number of perfect mixers, n	Nominal residence time of a perfect mixer, θ (min)
9	2.0	7.0	3.5	3.518	2.462	–	–
23	2.0	5.0	2.5	2.397	0.713	8.06	0.297
40	2.0	4.4	2.2	2.287	0.715	7.32	0.313
60	2.0	4.0	2.0	2.101	0.732	6.03	0.348

TABLE 6. Data for residence time distribution trials at different outlets of mixer and same speed.

Outlet	Distance from feed end, (cm)	Feed rate W (kg/min)	Material in mixer, G (kg)	Nominal residence time, G/W (min)	Mean residence time of observed distribution, μ (min)	Variance of observed distribution, σ^2	Number of perfect mixers,n	Nominal residence time of a perfect mixer, θ (min)
First	57	2.0	3.20	1.6	1.584	0.481	5.23	0.304
Second	82	2.0	4.65	2.3	2.498	1.008	6.19	0.404
Last	132	2.0	7.70	3.9	4.061	1.767	9.33	0.435

It is evident from tables 5 and 6 that the increases of speed and mixer length have opposite effects on the variable n which represents the corresponding number of perfect mixers. Increasing the speed reduces n while increasing the length increases n. Also the mean residence time of the distribution is decreased with increasing speed and is increased with increasing mixer length.

If, however, the mixer length L is devided by n and the quotient L/n is denoted by L', then we observe that L' is an increasing function of speed as well as mixer length. This can be clearly seen from the values given in table 7. From the same table it also can be seen that L' is not the same for the two trials with a speed of 23 r.p.m. and the second outlet of the mixer. Since these two trials were performed under the same experimental conditions of speed,

TABLE 7. L' ($= L/n$) as a function of speed and mixer length.

Second mixer outlet		Constant speed, 23 r.p.m.	
Speed r.p.m.	L'	Mixer outlet	L'
23	10.2 cm	First	11.0 cm
40	11.2 cm	Second	13.2 cm
60	13.6 cm	Last	14.1 cm

feed rate, and mixer length, but only differed in the type of mixing element, it can be concluded that the design of the mixing element affects the residence time distribution of the material. For the closely pitched mixing element (I) we found $n = 8.06$ while the same number was 6.19 for the widely pitched mixing element. Thus for the same operating conditions there is more longitudinal mixing for the widely pitched mixing element than for the closely pitched type. For the latter type only when the speed was increased to 60 r.p.m. was the number of perfect mixers equal to 6.03, and thus producing the same degree of longitudinal mixing which is produced by type II at a speed of 23 r.p.m.

The deviation of the observed distribution of 9 r.p.m. from the general form of the other six trials provides a good example of the kind of information that can be acquired from residence time distribution studies. In this trial sampling of the outflow stream was performed at every minute for the first three minutes, after which it was carried out at half minute intervals. By reference to figure 9 it will be observed that the distribution is not abnormal at the beginning, but tends to deviate at the end. In the latter part much more red than expected is found in the outflow stream. Such behaviour indicates the presence of a "dead space" or "dead spot" of the mixer in which the material is held for longer periods than required. This same feature of the mixing element (I) was also observed during the mixing studies reported earlier. However, the results here indicate that this dead space is only observable for very low speeds of the mixer.

A more appropriate graphical representation of the observed longitudinal mixing can be made by means of the F-diagrams as proposed by DANCKWERTS (1953b). This can be made by plotting the cumulative percentages against the dimensionless quantity $t/\bar{t}$ where $\bar{t}$ is considered to be the nominal residence time as determined by dividing the hold-up of the mixer by the mass flow rate. However, because the nominal residence time is not necessarily equal to the mean residence time of the observed distribution and in our case because of the inaccuracy of the measurements we shall use the mean residence time (μ) determined by the above mentioned method instead of $\bar{t}$. The results of the four trials with different speeds are given in figure 11.
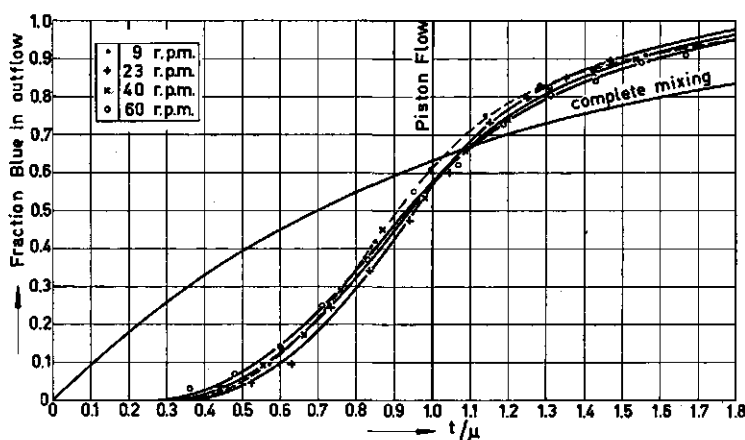

FIG. 11. F-diagram of the results reported in Fig. 9.

The main conclusions that can be drawn in this section are:
1. The methods which were developed for the investigation of the residence time distributions in continuous flow fluid systems, can also be applied for solids.
2. The mixer studied exhibits a substantial amount of longitudinal mixing which is influenced by the rotational speed of the mixing element and the length of the pitches of its component parts.
3. There seems to be no simple relationship between pitch length and the parameter n (number of perfect mixers). The only conclusion which we can draw is that increasing the speed or the length of the pitches increases L' ($=$ mixer length/n).
4. Under certain conditions, the residence time distribution can indicate the presence of dead spots in the mixer.

3.5. DISCUSSION AND CONCLUSIONS

The "SPAANS" mixer does not produce flow of material by steadily conveying it from one place to the other in the trough, but the flow is slug-wise in character. The quantity of material flowing through the mixer is not large, and the excessive turbulence caused by the mixing element produces quick but irregular and sudden improvements in mixture quality.

Because of the small quantity of material flowing through the mixer, sampling is not easy to perform. The mixture is easily disturbed and it is impossible to remove a sufficient number of samples from the same place and at the same time to acquire satisfactory information about the quality of the mixture. When only a small number of samples was used (as in the trial carried out with mixing element II) no meaningful conclusions could be drawn concerning the quality of the mixture. The mixing index indicated that mixing was complete. When the mixing element was removed before sampling no difference in performance could be observed at the different speeds. However fairly satisfactory results could be achieved by sampling in the presence of the mixing element and by collecting sufficient samples from replicates.

Still, sampling by the conventional thief results in excessive disturbance in the mixture. When the quantity of the mixture is large this disturbance is not of importance because the local differences in mixture quality are likely to be large, but when as in our case, the mixture is small in quantity and the local differences in mixture quality are not large, sampling by the conventional thief can lead to bad results. For these reasons a more accurate method of sampling might reveal differences which could not be detected by the procedure used in this study.

In spite of the moderate accuracy of the results, the main conclusions that can be drawn from the study are rather clear. The quality of the mixture at any place in the trough is determined by the speed and the design of the mixing element. Although mixing can be completed very quickly, almost instantaneously, by using high speeds, yet in practice the most appropriate speed to use would be determined by the characteristics of the material mixed and by economic considerations.

The observation that a mixing element with wider pitches of the spiral and the screw conveyor results in a more favourable distribution of the materials in the early part of the mixer, suggests the possibility of bringing about im-

provements in performance by changing the design of the mixing element. Perhaps, better performance can be achieved by a combination of different pitches on the same mixing element.

The length of the mixer does not seem to be of much importance concerning the effectiveness of transverse mixing achieved by the mixer. If the mixing element is not properly designed or a low speed is used for a certain material or a certain feed rate, transverse mixing would still not be good unless the mixer is made impractically long.

On the other hand, the length of the mixer is definitely related to the extent of longitudinal mixing taking place in the machine. Longitudinal mixing may and may not be desirable. If there are no fluctuations in the composition of the feed stream then the presence of longitudinal mixing is not of much advantage. However, when it is required from the mixer to smooth out fluctuations in the feed streams, then longitudinal mixing becomes of importance.

A continuous flow system which behaves as a perfect mixer is more effective in smoothing out fluctuations in the feed streams than a system which exhibits piston flow. Thus the ability of a mixer to smooth out fluctuations in composition of the feed stream can be increased by increasing the amount of longitudinal mixing. For our mixer it was found that this can be done by increasing the rotational speed of the mixing element and by increasing the length of the ribbon and the screw conveyor. The latter procedure also seems to eliminate "dead spots" from the mixer.

Furthermore, when the fluctuations in the composition of the inflow stream are known to follow a certain pattern and when the distribution of residence times of the system can be determined (as was succesfully done in this study), it is possible to compute the size of the mixer which is required to reduce the variance in composition of the inflow to a certain desired level in the outflow stream. For this purpose reference should be made to the article of DE BAUN and KATZ (1961).

From the general considerations treated above it follows that transverse mixing and longitudinal mixing are both of importance in continuous solids mixing. We showed that the former can be studied by the proper application of the general principles developed for investigating mixture quality and the latter by the methods used for residence time distribution studies. However, transverse mixing is accomplished in mixers of the type investigated so quickly, that details of the mixing process itself cannot be studied.

CHAPTER 4

EXPERIMENTAL CONTINUOUS MIXER
OF THE ROTATING CYLINDER TYPE

4.1. INTRODUCTION

In the previous chapter the work carried out on the "SPAANS" mixer was reported. The "SPAANS" mixer comprised a mechanical mixing element which rotated in a stationary U-shaped trough. The element produces flow by positive displacement of material along the helicoidal surface of a screw conveyor. At the same time mixing is performed by an outer spiral which acts in

order to disturb the smooth flow of material produced by the conveyor. For such a mixer we observed that the turbulence which causes the mixing is so large and the mixing is so rapid and non-uniform in pattern that it is impossible to define a simple relation between a mixing index and mixer length.

In this chapter we shall study another type of continuous solids mixers, namely the inclined rotating cylinder. This mixer employs a rotating wall in order to produce simultaneous flow and mixing of the material. It differs from the previous type because flow and mixing are determined by gravitational and frictional forces, while mechanical agitation is limited in character.

Diffusive mixing is considered to prevail in batch mixers of the rotating cylinder type under certain operating conditions. These conditions seem to be determined by the rotational speed, the diameter of the cylinder and the degree of filling.

By "diffusive mixing" we shall understand those mixing conditions under which the local concentrations of the different components of a mixture as measured by samples of practical sizes undergo gradual but not sudden changes during the mixing. Then the mean concentration in a certain volume element of any single component in a mixture of components that have no segregating tendencies, is a good index for the quality of the mixture. In a true binary diffusion process, the presence of a concentration gradient for one component in a certain direction requires the presence of an equal but oppositely directed gradient in the concentration of the second component. When the diffusion process is finished, the gradients are zero.

For radial diffusion to be the main mechanism of the exchange of matter in a continuous flow system, the flow pattern of the material through the system should not exhibit longitudinal mixing. The elements of material which enter the system at the same time, flow together through the system and finally leave the system at the same time.

Once a steady-state has been established in such a system the concentration of any component at any place within the system will be constant and will not vary with time if the feed is kept constant. The different components will be distributed in a definite pattern and sampling at the same place at different times will reveal the same composition but for the inevitable fluctuations due to random sampling errors.

A simple experimental continuous mixer for particulate materials which would most probably satisfy the requirements in the radial direction for diffusive mixing, is a simple rotating cylinder operated at a slight angle to the horizontal and in which the material to be mixed enters at one side and leaves at the other. For experimental purposes it is of course necessary to allow the entering streams to arrange themselves in a definite way such that mixing due to their entry remains negligible. Exchange of position of the particles of the different components of the accumulated mass would then take place by a process, which could adequately be described as diffusion in the radial direction. This chapter deals with the experimental results of a mixer which was designed in accordance with the general considerations above.

4.2. DESCRIPTION OF THE MIXER

The experimental mixer (see plate 3), consists of five main parts which will be referred to as: the supporting base, the variable-angle platform, the mixer-

drive assembly, the sampling arrangement, and the feed arrangement. Each of these parts will now be described in detail, and the different parts will be indicated by numbers which refer to those of plate 3.

The *supporting base* (1) consists of a six footed metal frame work with dimensions 0.85 × 0.80 × 1.90 meter, adequately reinforced by cross-bars for extra rigidity. It serves to carry the mixer at a sufficient height so that a weighing scale together with a collecting container of sufficient size to collect the material discharged during any trial, could be placed directly under the discharge opening of the mixer. The base supports the variable angle platform which in turn carries the remaining parts of the mixer.

The *variable-angle platform* (2) consists of a rectangular grid 0.85 meter wide and 1.90 meter long. It is connected to the supporting base at two points. The first of these occurs in the form of a pivoting hinge (3), located at approximately the center of gravity of the mass of the equipment lying above. The second connection takes place in the form of two screws moving within the openings of two slightly curved metal guides (4), located at the discharge side of the supporting base. The screws could be fixed to the guides by means of special nuts at any desired angle between zero and fifteen degrees with the horizontal. This arrangement allowed the angle at which the mixer is operated to be varied at will.

The platform is divided into two main parts. The first (2a) occurs at the feed end and is entirely covered by a metal plate which acts as a base for the mixer-motor assembly and the feed arrangement. The second part (2b) of the frame is uncovered and carries the special railing which accommodates the wheels of the sample-tubes tray (5). This same part also carries the special metal frames which hold the mixing cylinder in position (6).

The *feed arrangement* comprises two feed hoppers (7), each with a volume of about 0.007 cubic-meter, and two electro-magnetic vibrators (8), which deliver their streams to two separate funnels provided with plastic tubing to guide the material into the mixer. Each hopper is screwed to the ends of two separate metal bars located at appropriate positions. By means of these screws the height of the hoppers above the vibrators could be adjusted so that the depth of the material in the vibrator trough could be varied at will. Thus they could also be used to adjust the feed rate to the desired level.

Each electro-magnetic vibrator is carried on a special shelve provided with fixed metal sockets which closely accommodate the rubber legs of the vibrator in order to prevent accidental disturbance of the adjusted position of the vibrators.

The supporting bars of the feed hoppers as well as the shelves of the feed vibrators are carried on a common shelve whose angle could be varied, so that the position of the feed arrangement could always be kept parallel to the horizontal irrespective of the angle at which the mixer is operated.

Each vibrator feeds in a separate metal funnel which in turn opens via separate plastic tubing leading into the entry opening of the mixer. The two plastic tubings are strongly held in position by means of several coils of thread whose ends are firmly tied to the supporting frame of the rotating cylinder. This was found to be necessary since the tubes had to be of such length and to be placed in such a position so as to allow free flow of the material into the mixer without flooding of the tubes.

The *mixer-motor assembly* comprises an electric variable speed drive directly bolted in place to the covered part of the variable angle platform and a mixing element (9) in the form of a rotating cylindrical tube of "Plexiglass" (a polymetacrylate). The electric motor drives the cylinder by means of a small metal gear directly fixed to its rotating shaft. This gear drives a larger gear (10), fixed in position around the periphery of the mixing cylinder.

The mixing element, as mentioned already, is a cylindrical tube of "Plexiglass" in which material enters from one side and leaves at the other side. The discharge side of the tube is completely open, while to the feed entry side an extra disc of Plexiglass provided with an opening in the center, of 6 cm diameter, has been fixed to the internal wall of the cylinder. This disc serves as a barrier to the flow of material in the direction of the feed entry side. The feed enters the mixer by means of the two plastic tubings, already described, which slightly project into the cylinder through the central opening of the Plexiglass barrier disc.

The cylindrical tube is 126 cm long, with an internal diameter of 14 cm and a wall thickness of 0.5 cm. It is held in position by means of two metal rings (11) fitted to the periphery at 7.5 cm from both ends. The metal rings rotate in the grooves of plastic roller bearings fixed at appropriate positions to the supporting metal frames (6) which are in turn fixed to the variable-angle platform. At the discharge end, the Plexiglass tube was extended in length by a cylindrical metal piece of approximately 6 cm length. This metal piece carries a rubber

flap which acts as a precautionary element against starting the sampling mechanism at an inappropriate moment.

Material discharged from the mixer flows into a metal trough which directs it to the collecting container placed on a weighing scale. The quantity of material discharged during any period of time could thus be determined.

When the mixer was first tried, it was observed that the inner surface of the cylindrical tube was much too smooth to bring about the tumbling motion essential for mixers of the rotating cylinder type. In order to overcome this excessive slip at the wall, recourse had to be made to increase the roughness of the inner surface of the tube. This was performed by means of a piece of coarse sand paper tightly fixed to the tip of a metal rod, which was moved to and fro in longitudinal strokes along the entire inner surface of the cylinder.

The *sampling arrangement* comprises the sampling openings and their covers (12) which are automatically operated by means of an electro-magnet (13), the sample receiving funnels (14) which guide the removed samples to their separate containers and the sample collecting tubes held in place by means of a special holding tray (5). These different parts of the sampling arrangement are shown in greater detail in figure 12.
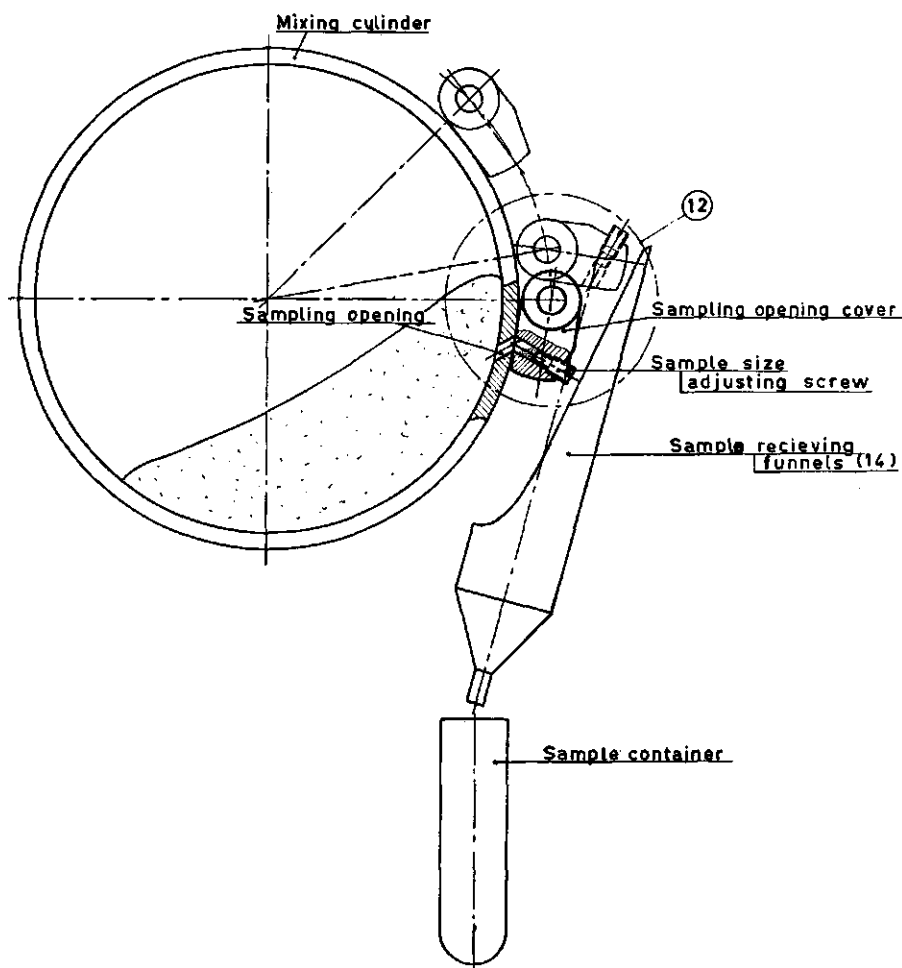

FIG. 12. Details of equipment for sample removing, the indicated numbers refer to the different parts described in the text.

For sampling purposes the cylinder was bored with inclined openings of 0.3 cm diameter at intervals of 5 cm starting at 12.5 cm from the feed end and ending at 18 cm from the discharge end of the Plexiglass cylinder. In total there were twenty such openings.

Each sampling hole had a separate metal cover. All such covers were tightly fixed to a common operating rod held in position by guides attached at adequate distances to the outer surface of the mixing cylinder. The covers were in the form of rectangular pieces of brass drilled to accommodate a screw at the place of contact with the sampling hole. The screw could be lowered or raised so that the size of the sample of material removed could be adjusted as desired. The whole assembly rotated with the rotating cylinder.

To the discharge end of the common rod operating the sampling holes' covers, a metal lever (15) was attached. This lever, and consequently the rod and the covers, was operated by a cam which moved forward and backward along the mixer axis by means of an electro-magnet. In its forward position the cam operated on the lever and the covers of all the sampling holes were simultaneously raised and samples could thus be collected. In its back-ward position the lever rotated freely with the cylinder and the covers remained closed on the holes. Two springs guaranteed that the sampling holes remained firmly closed, thus preventing any outflow of material.

The electro-magnet could be put on or off by means of a special electrical switch. When on, the action of the electro-magnet was regulated by means of two differently notched metal bars (16) attached to the side of the sample tubes tray. The ratio of the notches was five to one, thus as the closely notched bar progressed five positions, the other bar progressed only one position. Movement of the closely notched bar was determined by a lever which was in turn acted upon by a cam located on a cylindrical metal strip fixed to the outer wall of the cylinder. After every revolution of the cylinder the cam raised the lever and allowed the tray to advance one position. At the fifth position the widely notched bar acted on an electrical button which caused the electro-magnet to move its cam to the forward position and thus allow the removal of samples. Upon removal of the samples the cam operated by the electro-magnet moved backwards and the sampling holes were closed again. In this way the sampling schedule allowed the collection of twenty samples simultaneously every five revolutions.

Every sampling hole delivered its material to a separate sample receiving funnel (14) which guided the particles to the sample tubes located below. The sample tubes were ordinary test-tubes of 1.8 cm diameter cut to a suitable height to allow free movement of the tubes under the funnels. The tubes were held on an aluminium tray bored at adequate intervals so that the funnels' openings corresponded with the center of the tubes after the tray moved five positions, as determined by the notched bar already described. The tray is provided with four wheels which run on a special railing, and it moves under the influence of a weight hanging at the end of a plastic rope, connected to the tray and passing over a pulley especially placed to facilitate movement of the rope.

4.3. EXPERIMENTAL MATERIAL

The same material as used previously with the "SPAANS" mixer was used here: namely closely sized sand particles with diameters ranging between 0.4 mm and 0.6 mm. The sand was coloured blue and red with the same pigments as before viz. Methylene Blue and Waxoline Rhodamine (I.C.I.). Material from the different trials was not discarded but was used again after washing it with water to remove the red pigment and then colouring all with blue. The material was then dried and sieved before use. For the red sand, it was necessary to dry, sieve, colour, dry and resieve new batches of sand every time. Sufficient material was always prepared in advance so that the experimental work could proceed without delay. The sand had the physical properties reported earlier.

4.4. PRELIMINARY INVESTIGATIONS

The object of these investigations was to test the experimental equipment and to acquire more information about the sampling technique and its adequacy for further studies.

From the introduction and the description of the mixer it becomes obvious

that the sampling procedure to be used here differs from those which have been usually used in mixing studies. The conventional and widely accepted procedure of using a sampling thief to collect the samples, will be replaced by a sampling method which removes samples from only a single point of the mixture. For this purpose the samples will be removed from the layers of material near to the rotating wall by the procedure described in section 4.3. The object from such a sampling procedure was to avoid complicating the design of the mixer and to try out a sampling method which does not cause excessive disturbance to the mixture.

To carry out any trial it was first necessary to adjust the mixer to the operating conditions to be investigated. Adjustment of the speed of rotation was straight forward and no special precautions were needed to get a constant speed. The same holds for the angle at which the mixer was to be operated. The procedure followed for this purpose was to take measurements of distances and computing the angle made with the horizontal from trigonometric relations. The single operating variable which required extreme care in its adjustment was the feed rate. At this stage of the work it was not as yet required to adjust the feed rate to a certain prescribed level, and for the purpose of the investigations carried out here, it was sufficient to determine the quantity of material delivered by each vibrator in a certain period of time. For this purpose the mixer was allowed to rotate and one of the vibrators was allowed to deliver its material. After some time when it was considered that no more accumulation of material was taking place in the cylinder and thus a steady-state has been established, the quantity of material discharged during a certain period of time was determined. The same procedure was repeated for the second vibrator and the feed rate was then considered to be the quantity of material delivered per unit time.

The sample size was also adjusted so as to contain about 160 particles. This was done by letting the mixer operate and collecting and counting several samples from each sampling location. The screw in the sample holes covers was then raised or lowered according to whether it was necessary to increase or decrease the size of the sample under consideration. After the first adjustment, another series of samples was collected and counted, and according to the results the final adjustment was made.

The experimental work was started by a trial involving a speed of 5 r.p.m. and a feed rate of 15.2 g/sec (sum of blue and red in a proportion of about 1 : 1). The mixer was operated at an angle of four degrees with the horizontal.

After the operating conditions were adjusted, the trial was started by allowing the mixer to rotate and the vibrators to deliver their feed stream. This was allowed to proceed for some time, during which the electro-magnet was kept out of function. The material discharged was then determined at several short periods of time until it was observed that the feed rate was more or less constant at the desired level. Once the feed rate was observed to be constant the mixer was operated for several more minutes before sampling was started.

The electro-magnet was then switched on, and the sample tube tray was allowed to move forward. Every five revolutions twenty samples were removed automatically and this was allowed to proceed until in total only 15 samples per sampling location were collected, although the tray could accept 17 samples from any one place. From the 15 samples only the last ten samples were used for analysis. The choice of ten samples was only made to decrease the amount of work required for the analysis, and although this number later proved to

be insufficient to get a high degree of accuracy, no effort was made to increase the number of samples because the amount of work required for sample analysis would have become prohibitive.

During the trial the feed hoppers were kept completely and continuously full. In order to prevent any particles from falling into the sample tubes during the filling of the feed hoppers, a sheet of paper was used to cover the tubes directly after receiving the samples. When sampling was over, the electro-magnet was switched out and the mixer stopped. The tubes were then removed from the tray and arranged in the order of their removal in a wooden rack especially made for the purpose.

For analysis each tube was removed in turn. The contents were spread over the surface of a white piece of paper by gentle tapping and in a more or less straight line in order to facilitate counting. The number of red and blue particles were then counted by the aid of a magnifying glass and from the counts the proportion of blue sand in the samples was determined and the results were recorded in tabular form. For this initial trial the proportion of blue sand in the ten samples of the first twelve sampling locations was determined.
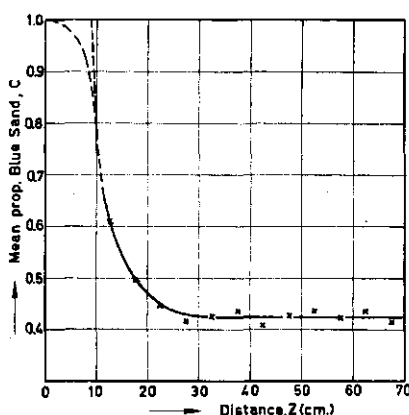

FIG. 13.
Mean proportion blue sand against distance at which sampling was performed. Mixer operated at four degrees with the horizontal. Rotational speed 5 r.p.m. and feed rate 15.2 g/sec.

The mean proportion of blue sand (C) was plotted against (z) the distance in centimeters from the feed end, at which they were removed. The results are given in figure 13. From this figure it can be seen that the quantity of blue sand at the wall of the mixer is high in the early part of the mixer and quickly decreased to a more or less constant value. It required approximately 25 cm of mixer length to reach this constant value. It is also seen that the composition of samples at distances of less than 12.5 cm could not be determined because the boring of the sampling holes was started at this distance. Thus the true shape of the curve in this early part of the mixer remains undetermined.

The quickness by which the sample composition reached a constant value, raised however the question of whether this was entirely due to mixing brought about by the mixer itself, or whether it was due to the mixing brought about by the intermingling of the two feed streams during their entry into the tube. Visual inspection revealed that indeed an appreciable amount of mixing does take place merely due to the fact that the two feed streams during their entry actually fall upon each other in an irregular way. Thus blue and red particles

are already mixed to a high degree before they start their radial rotation in harmony with the regular pattern of motion expected for mixers of the rotating cylinder type.

Since it is the object of the present study to investigate the mixing brought about by the mixer itself and not that which is due to the method of entry, it became necessary to modify the mixer in this early part so as to avoid this entry mixing as far as was practicable.

Accordingly two technical changes were made for this purpose. In the first place a metal piece 9.0 cm high and 10.0 cm long was placed between the ends of the plastic tubes projecting into the cylinder. The lower edge of the metal piece was placed at approximately 0.3 cm above the wall of the cylinder rotating beneath it. This was done to separate the two entering streams from each other. The effect of this modification was that the blue particles falling on one side of the metal piece moved from below the piece under the influence of the rotating wall, and collected on the other side of the partition to form a layer of entirely blue particles at the wall. At the same time the red particles tended to fall above the blue layer. The two layers gradually accumulated to a maximum possible height determined by a combination of feed rate, speed and angle.

The second modification made to minimize the effect of entry mixing was to polish the first 10.0 cm of the inner surface of the cylinder very smoothly. By this modification it was hoped to increase the degree of slip at the wall so that the material accumulated without undergoing an appreciable degree of rotation. This precaution succeeded to fulfill its objective up to a certain extent. During the early phases of the accumulation of the material in this part of the mixer, it was observed that indeed the material at the wall tended to exhibit an appreciable amount of slip without acquiring any apparent degree of rotation. However, by the time the accumulation has reached its maximum constant value the rotation seen in mixers of the rotating cylinder type was obvious. Since the material at the wall was entirely blue, the early part of the mixer showed only blue particles with the red, except for the first one or two centimeters, being completely hidden by the blue.

With these modifications completed two consecutive trials involving 7 and 9 r.p.m. were performed. The vibrators were adjusted to deliver about 15.2 g/sec in the proportion of 0.6 blue to 0.4 red, and the mixer was operated at an angle of about three degrees with the horizontal.

The experimental procedure used for these two trials followed the same lines as that described earlier. In addition a further check on the feed rate was made
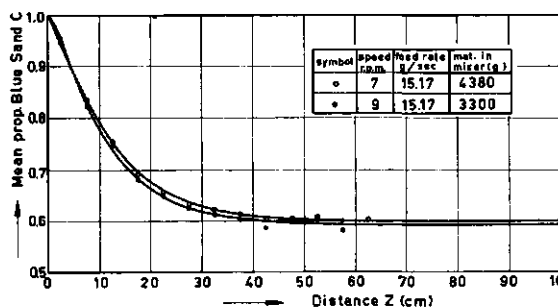

symbol	speed r.p.m.	feed rate g/sec	mat. in mixer(g)
○	7	15.17	4380
●	9	15.17	3300

FIG. 14.
C vs. distance for the indicated rotational speeds and feed rate. The point of zero distance corresponds with 10 cm from feed end.

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and the quantity of material in the mixer was determined. The results of these two experiments are reported in figure 14. This graph differs from the previous one in that the mixing process was considered to start at 10 cm from the feed end. This decrease in distance was made because it was assumed that no mixing took place in the first 10 cm of the mixer because of the modifications described earlier. As seen from the graph twelve points for the trial with 9 r.p.m. and 13 points with the trial of 7 r.p.m. have been reported. The reason is that the analysis of the samples was continued only up to the point where the mean proportion of blue sand tended to become more or less constant. Extra points were assumed not to contribute much to the accuracy of the results. In any case, sufficient points are available to give a fairly good idea about what can be expected if further distances were analysed.

By comparison with figure 13 certain definite changes can be seen to have taken place. It is obvious that the constancy of sample composition has been delayed to more than fourty centimeters. The first sampling locations showed mean compositions very near to hundred per cent, which indicates that the modifications introduced fulfilled their objectives reasonably well. Furthermore, the graph clearly shows that the first 10 cm of the mixer, which have been left out of consideration, do not contribute much to the mixing process.

From figure 14 it can also be seen that the experimental points can be represented by a smooth curve and that these points closely fit the curve except at the final part where greater fluctuations are seen to take place. It is further evident that the curves for the two speeds studied did not coincide but actually show a certain small but definite difference. The curves also indicate that the final constant composition of the mixture is not the same for both trials. Accordingly the plot does not allow the drawing of any conclusions concerning the effect of speed on the mixing process. What they do indicate, however, is, as was expected, that the mixing process is one which tends to an equilibrium state.

The residence time distribution of the material in the mixer was also determined. As with the "SPAANS" mixer the procedure used here involved a stepwise disturbance and the determination of the distribution of residence times from the composition of the outlet stream. Only one trial was performed. It was carried out at a speed of 10 r.p.m. and a feed rate (W) of 13.3 g/sec. The quantity (G) of material accumulated in the mixer was found to be 2590 g. Thus the mean (nominal) residence time $(\bar{t})$ could be determined $(\bar{t} = G/W)$ to be 194 seconds.

First red sand was allowed to flow through the mixer and several measurements on feed rate were made. When the amount of material discharged was found to be constant at 13.3 g/sec and thus indicating that no accumulation was taking place, the stream of red was shut off and that of blue turned on. At this same instant the time of flow was registered, and the zero time of the measurements was considered to be the time of change-over from the red to the blue stream.

From the stream of material leaving the mixer approximately ten grams of material were removed at definite time intervals by quickly moving a small beaker glass through the outflowing stream. This sampling procedure was started at 2 min 35 sec from the time the stream of red was replaced by that of blue. It was not necessary to take earlier samples because the boundary between red and blue in the flowing mass was clearly visible. Other samples were

removed at 2 min 50 sec, 3 min 8 sec, 3 min 20 sec and 3 min 35 sec respectively, thus at approximately 15 sec intervals except for sample three which was removed at 18 sec from the time of sampling of the previous one. All sampling times are of course approximate. Sampling was started just before the 15 sec interval and ended just after this period.

Each of the five batches of material removed as described above was well mixed and considered to represent the exit stream at the time of sampling. After mixing, these batches were sub-sampled by means of a sampling thief. From every initial batch, five samples of approximately 180 particles each were collected and analysed. The proportion of blue sand was determined from counts as usual and the mean value of every five samples was taken to represent the composition of the exit stream at the time of sampling. The five mean proportions thus derived were plotted against the dimensionless quantity $(t/\bar{t})$ in figure 15.
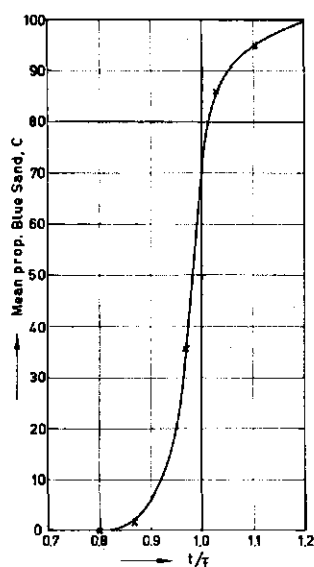

FIG. 15.
F-diagram for the rotary cylinder operated at 10 r.p.m. and a feed rate of 13.3 g/sec.

From this it is seen that the experimental points can be represented by a smooth curve which deviates from the vertical line representing piston flow. The deviation is, however, not great and the excessive steepness of the experimental curve indicates that no appreciable degree of longitudinal mixing takes place. Actually the experimental curve resembles those which have been reported for fluids flowing in a turbulent regime [DANCKWERTS (1953b)].

4.5. MATHEMATICAL REPRESENTATION OF THE RESULTS

It has already been remarked that the plot of composition against distance for different speeds did not allow the drawing of any conclusions concerning the effect of rotational speed on the mixing process. Since it is the object of this study to know more about the operating characteristics of the mixer it is

necessary to have some suitable quantitative measure for the performance of the mixer under different operating conditions.

Assuming a diffusion analogy, at first thought the most relevant approach to the problem would be to treat the process quantitatively along lines similar to those used for fluids. Unfortunately it is not easy to adopt such a procedure. The difficulties involved will become clear from the discussion given below.

Of the three possible mechanisms of mixing that have been postulated, we assume that only those of diffusion and convection are important in a mixer of the type under consideration. Convective mixing will be of importance in the first part of the mixer but after the process has proceeded for some time the rate of diffusion will be the controlling factor in the process. In order to simplify the problem somewhat we shall make the assumption that only a small portion of the transfer is due to convective mixing, so that the convection rate can be included in a diffusion rate.

Under these assumptions, the problem at hand can thus be stated as one which deals with the radial transfer of mass by the process of diffusion, within the mass of a uniformly moving substance, the cross-section of which is a circular segment whose area is determined by the conditions under which the mixer is operated.

When the process is considered analogous to the flow of fluids in conduits, by making a material balance around a differential element of volume, it can be shown that the process of transfer can be represented by a partial differential equation of the following form:

$$v \frac{\partial C}{\partial z} = D \left(\frac{\partial^2 C}{\partial r^2} + \frac{1}{r} \frac{\partial C}{\partial r} \right)$$

where $C(r, z)$ is the concentration of a certain component of the system, D is the coefficient of diffusion, v is the flow velocity assumed to be constant, and z is the distance in the direction of flow. In this equation the effect of axial diffusion is considered negligible, and cylindrical coordinates are used.

A solution of this equation for our boundary conditions is not known. However it is possible to illustrate qualitatively the influence of the operating variables by considering the idealized case where the circular segment is replaced by a sector, in which the two materials to be mixed are initially arranged as in figure 16.
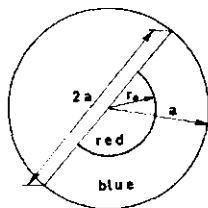

FIG. 16.
Schematic illustration of the initial boundary conditions of the system for which the diffusion equation has been solved.

For this special case the concentration of blue at the entrance is described by:

$$C_0(r) = \begin{cases} 0 & \text{for } 0 \leqslant r \leqslant r_0 \\ 1 & \text{for } r_0 \leqslant r \leqslant a \end{cases}$$

and the solution for the appropriate boundary conditions can be shown to be:

$$C(r, z) = \left(1 - \frac{r_0^2}{a^2}\right) + \frac{2r_0}{a} \cdot \sum_{n=1}^{\infty} e^{-\frac{D \cdot \beta_n^2 \cdot z}{a^2 \cdot v}} \cdot \frac{J_1\left(\frac{r_0}{a} \cdot \beta_n\right)}{\beta_n} \cdot \frac{J_0\left(\frac{r}{a} \cdot \beta_n\right)}{J_0^2(\beta_n)}$$

where J_0 indicates a Bessel function of the first kind and zero order,
  J_1 indicates a Bessel function of the first kind and first order,
  βn are the positive zeros of J_1,
and the other symbols have the same meaning as given previously.

The term $\left(1 - \frac{r_0^2}{a^2}\right)$ describes the situation of complete mixing at the entrance of the tube, while the subsequent terms are corrections for the degree of deviation from such a picture. Actually this term represents the fraction of the total volume of the system occupied by the blue particles, and it thus represents the proportion of blue present at equilibrium. For large values of z, the equation can thus be reduced to the following form:

$$\Delta C = e^{-\frac{D \cdot \beta_1^2 \cdot z}{a^2 \cdot v}} \cdot \frac{2r_0}{a} \cdot \frac{J_1\left(\frac{r_0}{a} \cdot \beta_1\right)}{\beta_1} \cdot \frac{J_0\left(\frac{r}{a} \cdot \beta_1\right)}{J_0^2(\beta_1)}$$

From this it can be seen that deviation from equilibrium can be represented by an exponential function involving distance, area of transfer and the linear velocity of the system.

Because of the reasons given above and because of the absence of a better alternative, we decided to try to represent our results by the following simple expression:

$$\frac{\Delta C}{\Delta C_0} = e^{-\frac{D \cdot \rho z}{W}}$$

where $\Delta C = C_i - C_{eq} = $ the difference between the proportion blue sand (C_i) at distance i in the mixer and that which is present at equilibrium (C_{eq}).

$\Delta C_0 = $ the value of ΔC at zero distance
$z \quad = $ distance in centimeters along length of mixer
$W \quad = $ mass flow rate g/sec
$\rho \quad = $ bulk density of the material at rest g/cm^3
$D \quad = $ a constant representing the rate of transfer and thus the rate of mixing for the conditions under which the mixer is operated, cm^2/sec
$e \quad = $ base to the natural logarithms.

When applying this equation one is faced with the difficulty of finding the best value for the equilibrium concentration, C_{eq}. In batch mixing this difficulty is not present because the quantities of blue and red sand are exactly known. The same would have also been the case here if the feed mechanism was exact and did not exhibit any fluctuations. With the equipment used in these studies this did not prove to be the case, and the quantity of material delivered per

unit time could not be taken as a reliable estimate of the equilibrium concentration C_{eq}.

A reliable estimate could of course be acquired by analysing many points in the constant part of the mixture and taking the average value of these points to represent C_{eq}. This procedure was, however, not adopted because it would have entailed an excessive amount of work for the analysis. In place of such a procedure it was decided to determine C_{eq} by extending the fitted curve to a point where the proportion of blue sand could more or less be considered constant. This estimated constant value of the fitted curve was taken to represent C_{eq}.

Another source of error in applying the proposed equation is using the bulk density of the material at rest in place of its bulk density in flow. The former quantity is usually considered to be higher than the latter. As far as this study is concerned the error introduced by making this approximation is of minor importance, since we are only concerned here with finding some suitable quantitative expression which allows the making of reasonable conclusions about the operating conditions of the mixer.

The simple equation gives a straight line if a plot of $\Delta C/\Delta C_0$ versus $\rho \cdot z/W$ on semi-logarithmic paper is made. The slope is negative because $\Delta C/\Delta C_0$ decreases with distance along the length of the mixer. Application of these concepts to the results of the two trials involving 7 and 9 r.p.m. gives the graphs of figure 17.
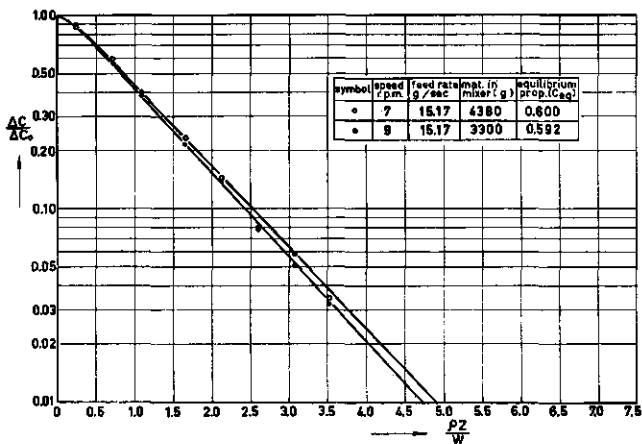

symbol	speed r.p.m.	feed rate g/sec	mat. in mixer(g)	equilibrium prop.(C_{eq})
•	7	15.17	4380	0.600
•	9	15.17	3300	0.592

FIG. 17.
Log$\Delta C/\Delta C_0$ vs. $\rho z/.W$
Mixer rotated at indicated speeds.

From the figure it is seen that the graphs are initially curved but quickly tend to the linearity we hoped for.

Another point of interest which is clearly evident from the graphs, is the fact that the process can only be followed up to a certain point. This reflects the inability of ten samples to detect further differences in concentration after this point. To be able to follow the process further, statistical considerations show that many samples would have to be analysed.

The plot of figure 17 also indicates that speed of rotation seems to have some effect on the rate of mixing. However, because the difference is small and the scatter of the points about the fitted line is large no real meaning can be given to the difference.

Although the results reported above were encouraging yet they were too few to have any real significant value. It thus became desirable to investigate the effect of speed further. For this purpose it was necessary to adjust the feed rates very accurately. This caused a lot of trouble and required much time and work to study the operating characteristics of the available vibrators.

The procedure which was finally adopted involved letting the vibrators work for at least two hours while empty before every trial. They were then stopped and filled with material and the required feed rate was adjusted. During every trial the feed rate was checked. If the results were satisfactory the samples were analysed. If an excessive deviation was observed the samples were discarded and the trial repeated.

Another problem was that by continuous use of the cylinder its inner surface lost much of its initial roughness. However, it was sufficiently rough to prevent the excessive slip observed when it was new. The effect of surface roughness has been investigated and will be dealt with later.

4.6. Investigation of several operating characteristics of the mixer
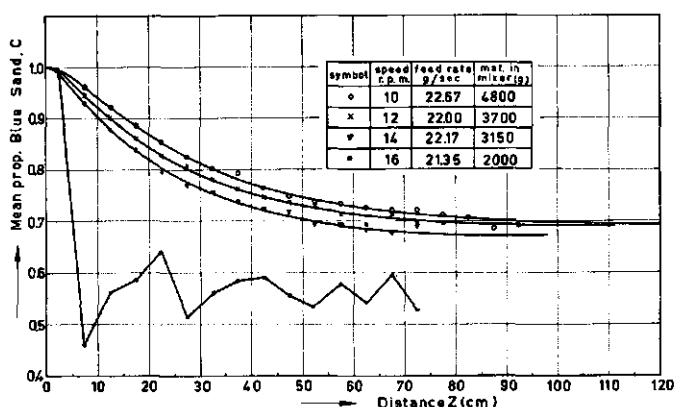
A systematic study of the possible variables would cost such a tremendous amount of time that we were forced to restrict ourselves to a few exploratory experiments. Furthermore, the simple design of the machine investigated did not allow the study of a great many variables. Of the several possible factors which can influence the performance of the machine as a continuous solids mixer only rotational speed, feed rate, wall roughness and the relative proportions of the materials mixed were briefly studied. The angle of inclination was kept constant at approximately 3 degrees.

Speed of rotation. It was observed that, for a constant feed rate the rotational speed of the mixer could only be varied within a limited range. At a certain low speed it was found that the material flowing through the cylinder tended to accumulate to a height which allowed it to overflow through the inlet opening. On the other hand, at a speed not much higher than the previous one, the material did not accumulate in sufficient quantity to allow the characteristic tumbling motion of the particles to take place. Within the limits of such a narrow range of possible operation, only a few closely spaced speeds could be investigated.

It is clear that one of the effects of speed is that it determines the quantity of material accumulated in the cylinder. The probable pattern which the blue and red particles tend to follow in arranging themselves in the mixer after their entry has been dealt with in section (4.4). There, it has been indicated that the particles after their entry tend to collect on one side of the cylinder under the influence of the rotating wall. For a certain material, accumulation will proceed until a maximum possible height which will be determined by the conditions under which the mixer is operated. The accumulated layer will finally assume an inclined position.

The way the mixing process is affected by speed will be discussed from the results of four trials. The trials were carried out at speeds of 10, 12, 14 and 16 r.p.m. respectively. Herewith an attempt was made to keep the feed rate and thus the relative proportions of blue to red at the same constant value by the procedure previously explained. The determined values are given in the tables accompanying figures 18 and 19. From these it will be observed that the feed

FIG. 18. C vs. distance. Experimental results for different rotational speeds.

symbol	speed r.p.m.	feed rate g/sec	mat. in mixer(g)
◇	10	22.67	4800
×	12	22.00	3700
▼	14	22.17	3150
▪	16	21.35	2000

rate as well as the equilibrium proportion were not constant from trial to trial. However, the observed discrepancies are of minor importance as far as the present discussion is concerned and will at the moment be left out of consideration.

In figure 18 the results of the sample analysis of the four trials are given. By reference to the figure it will be observed that for the first three speeds the experimental points can be described fairly well by a smooth curve. The same does not hold for the results of the fourth trial which involved a speed of 16 r.p.m. For this trial the speed was too high so that no appreciable radial movement of the particles took place. Evidently not much mixing has taken place up to 70 cm of mixer length, at which point the sample analysis was stopped. The proportion of blue sand at the different points fluctuates between high and low values in an erratic manner. The fluctuations also indicate that the composition of the different points does not follow any definite pattern and merely depends on the proportion of blue to red at the moment of sampling.

The sudden change in composition between the first two sampling points can also be explained. During the trial it was observed that the layer at the wall was still blue. However, above this layer, a layer of red could also be seen. The red layer remained above the blue layer until a short distance after
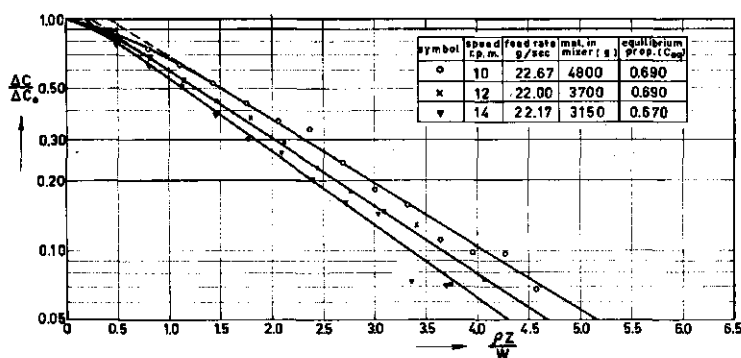

symbol	speed r.p.m.	feed rate g/sec	mat. in mixer(g)	equilibrium prop.(C_{eq})
○	10	22.67	4800	0.690
×	12	22.00	3700	0.690
▼	14	22.17	3150	0.670

FIG. 19. Log $\Delta C/\Delta C_0$ vs. $\rho z/W$. Mixer rotated at indicated speeds.

the partitioning metal piece, whereupon it curved downwards toward the bottom of the accumulated material, thus accounting for the sudden increase in red between these two sampling points.

The difference between the regular pattern observed for the first three trials and the erratic behaviour of the fourth is also reflected in the type of surface observed in the two cases. This can be seen in the pictures of plate 4. In picture (1) of this plate, the surface is complete and represents that surface which exists when the operating conditions of the mixer allow the characteristic tumbling motion of the particles to take place. It is the type of surface required for mixing to take place in mixers of the type investigated. However, when the feed rate is too low or the speed is too high, the continuous and complete surface breaks up into two distinct planes as is clearly seen from picture 2 of the plate. The presence of such a surface reflects the failure of the machine to establish its mixing mechanism under the operating conditions prevailing.

With the equilibrium proportions determined from figure 18 a plot of $\Delta C/\Delta C_0$ vs. $\rho z/W$ for the trials with 10, 12 and 14 r.p.m. has been made in figure 19. The curves reveal the same general features of the curves reported earlier in section (4.5).

However, the lines are more clearly separated and the different slopes of the linear portion of the curves indicate that speed exercises some effect on the mixing process. To show the relation existing between speed and the slopes of the curves, the value of D was determined for every curve by measuring the slope of the linear part and multiplying by 2.303 to convert from common to natural logarithms. A plot of D against speed is given in figure 20. This shows that D increases with increasing speed. For the range of speeds studied, the relationship between D and r.p.m. seems to be linear.
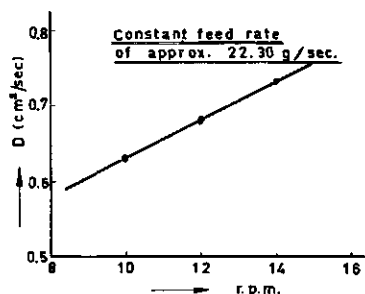

Fig. 20
Rate of mixing vs. rotational speed. Values of rate of mixing are obtained from the slope of the linear portions of Fig. 19 as described in the text.

Conclusion. When the previous results are considered together, it can be concluded that in mixers of the type considered, increasing the speed of rotation will continuously increase the rate of mixing up to a certain limit after which further increase in speed will result in the failure of the machine to mix properly.

Proportion and Surface Roughness. Two pairs of trials were performed to study the effect of these factors. Apart from the degree of roughness of the cylinder and the proportion of blue to red, the other variables were held constant within the practical limits possible. Thus all four trials were carried out at a speed of 14 r.p.m. and care was taken to keep the feed rate close to the average value of 22.3 g/sec. The exact values as well as the amounts of material accumulated in the mixer in every case are given in the tables accompanying the graphs of the results.

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In order to adjust for proportion, the necessary quantities of blue and red were computed from the total feed rate of 22.3 g/sec. Each vibrator was then adjusted in turn to deliver the quantity required. The first of the two vibrators was, however, only roughly adjusted to deliver a quantity of material approximately near to that required. The second vibrator was then adjusted to deliver a stream of material which together with that of the first brought the total feed rate to 22.3 g/sec. The work to produce a constant feed rate was so tedious that no effort was made to get a definite proportion of blue to red from trial to trial.

Each pair of trials was performed at a level of surface roughness of the cylinder, different from that at which the other pair was carried out. The first pair of trials, which will be indicated in the graphs by I, was performed with a cylinder surface which had lost much of its initial roughening due to continuous usage of the machine. The second pair of trials which shall be indicated by II was carried out after all the trials reported in this study were performed. For the purpose of these trials, the inner surface of the cylinder was roughened again by longitudinal stroking with a piece of coarse sand paper in the manner previously described. Thus the term degree of roughness referred to here, is only qualitative in character and no effort was made to express the level of roughness in a quantitative way.

The results of the four trials are reported in figures 21, 22, 23 and 24. As usual the plot of figure 21 was used to determine the equilibrium concentrations necessary for plotting figure 22. It is obvious from the graphs of figure 21 that the equilibrium concentrations widely differed from trial to trial.

In figure 22, the pair of curves at the top represents the trials performed at a low level of surface roughness I and that at the bottom represents the trials carried out at a high level of surface roughness II.

Conclusions. From figure 22, it can be seen that at the same level of surface roughness, the linear parts of the curves run parallel to each other. Thus the D values are equal and the mixing rate does not seem to be affected by proportion. When ΔC instead of $\Delta C/\Delta C_0$ was plotted against $\rho z/W$, it was found that the results of each pair of trials could be represented by a single line as is evident from figures 23 and 24. Thus apart from the initial part of the curve it seems that the rate with which equilibrium is reached is the same for a high as for a
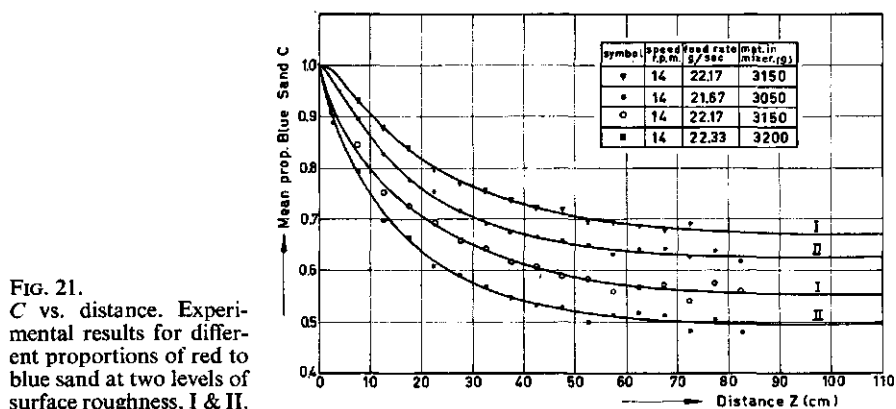

FIG. 21.
C vs. distance. Experimental results for different proportions of red to blue sand at two levels of surface roughness, I & II.
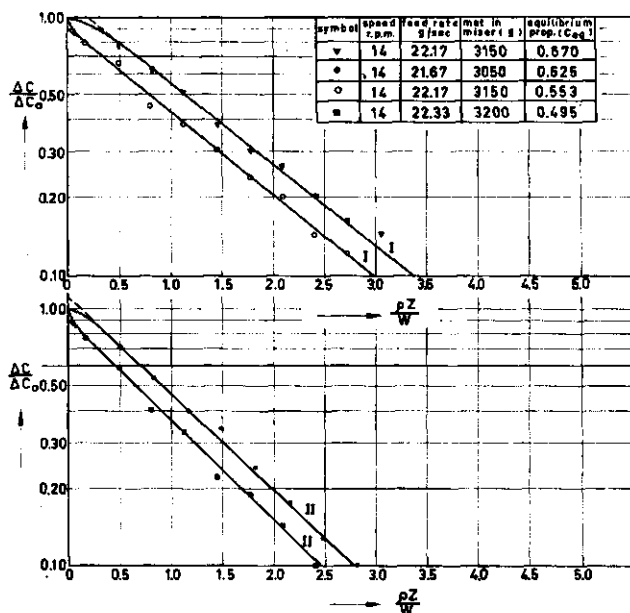
FIG. 22.
Log $\Delta C/\Delta C_0$ vs. $\rho z/W$ for different proportions of red to blue sand. Upper graph at "low" level of surface roughness (I) and lower graph at "high" level of surface roughness (II).

symbol	speed r.p.m.	feed rate g/sec	mat in mixer(g)	equilibrium prop.(Ceq)
▼	14	22.17	3150	0.670
●	14	21.67	3050	0.525
○	14	22.17	3150	0.553
■	14	22.33	3200	0.495

FIG. 23.
Log ΔC vs. $\rho z/W$ for different proportions of red to blue sand.

symbol	speed r.p.m.	feed rate g/sec	mat in mixer(g)	equilibrium prop.(Ceq)
▼	14	22.17	3150	0.670
○	14	22.17	3150	0.553

symbol	speed r.p.m.	feed rate g/sec	mat in mixer(g)	equilibrium prop.(Ceq)
●	14	21.67	3050	0.525
■	14	22.33	3200	0.495

FIG. 24.
Log ΔC vs. $\rho z/W$ for different proportions of red to blue sand.

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low proportion of blue to red. The only evident difference between mixing materials of different proportions seems to be reflected in the form of the early part of the plot. The tendency to linearity seems to be reached much quicker for proportions near 1 : 1 than for wider proportions of blue to red.

The two pairs of trials had, however, different slopes. The D values determined for each pair were found to be 0.73 and 0.90 cm²/sec respectively. This clearly demonstrates that surface roughness affects the rate of mixing. A higher level of surface roughness results in a higher rate.

Feed Rate. For the same reasons stated in the discussion concerned with the effect of speed of rotation, the feed rate could not be varied within wide limits. Adjustment of the machine to study this effect was greatly facilitated by the fact that proportion had no effect on the rate of mixing. The results of five trials will be used· to illustrate in which way the feed rate affects the mixing process.

The experimental procedure followed was relatively simple. All that was required to carry out a new trial was to adjust the vibrators to deliver a total quantity of material different from that delivered per unit time in the other trials. No effort was thus made to get the same proportion of blue to red in the different trials. As will be observed the equilibrium concentrations for the different trials varied to a great degree from trial to trial.

Of the five trials three were carried out at 10 r.p.m. while the other two involved a speed of 12 r.p.m. The trials with a speed of 10 r.p.m. will be discussed first.
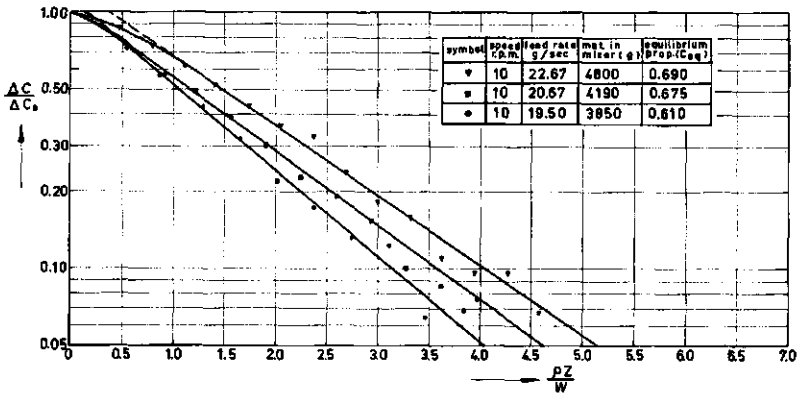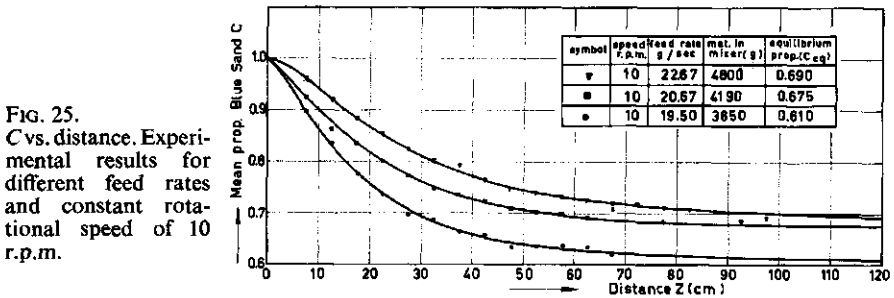
Fig. 25.
C vs. distance. Experimental results for different feed rates and constant rotational speed of 10 r.p.m.

Fig. 26. Log $\Delta C/\Delta C_0$ vs. $\rho z/W$ for different feed rates as indicated.
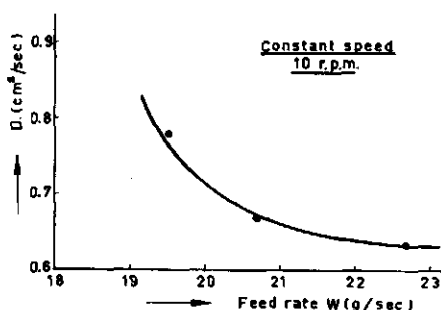
FIG. 27.
Rate of mixing vs. feed rate. Values of rate of mixing are obtained from the slope of the linear portions of Fig. 26 as described in the text.

Figures 25 and 26 represent the usual way of expressing the results. From figure 26 it is evident that the feed rate has an appreciable effect on the performance of the machine. The relation between the D values and feed rate can be observed from figure 27. A high feed rate results in a low value and the reverse is true. The rate of mixing therefore decreases with increasing feed rate. The decrease is at first fast but quickly tends to diminish in magnitude. It seems that at feed rates greater than 23 g/sec for a speed of 10 r.p.m., the D values will eventually tend to a constant minimum value, the other operating

FIG. 28.
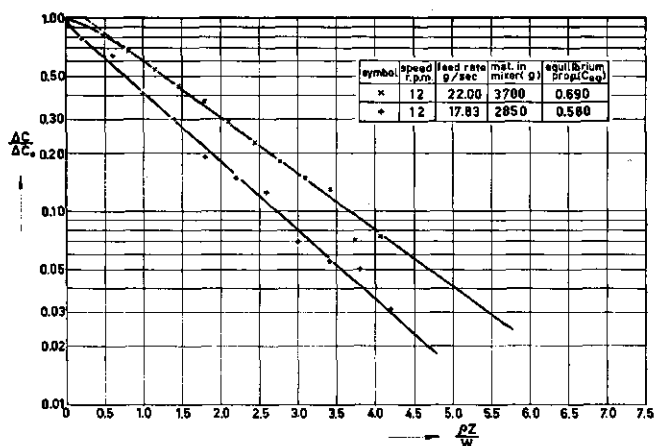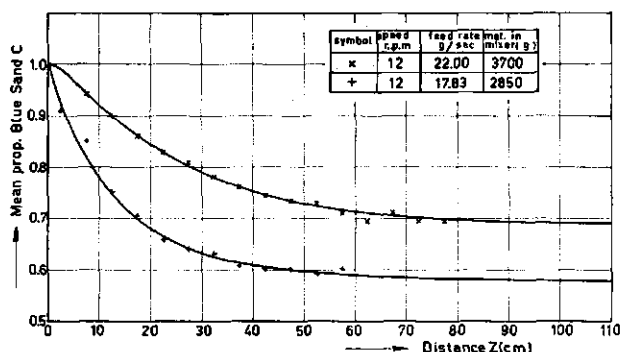C vs. distance. Experimental results for different feed rates and constant speed of 12 r.p.m.

FIG. 29.
Log $\Delta C/\Delta C_0$ vs $\rho z/W$ for different feed rates as indicated. Mixer rotated at constant speed of 12 r.p.m.

conditions being kept constant. On the other hand as the feed rate decreases the rate of mixing increases. Although it is not evident from the plot of figure 27, the previous findings indicate that at a certain level of wall roughness there will also be an upper limit to the increase in D values.

The results of the two trials with 12 r.p.m. are given in figures 28 and 29. The clear difference in the slopes of the curves which involve feed rates of 22.0 g/sec and 17.8 g/sec also confirms the findings reported above.

Conclusion. From the previous results it can be concluded that mixing occurs at a faster rate for low feed rates than for high feed rates, the speed being constant.

4.7. DISCUSSION AND CONCLUSIONS

In the previous pages we have generally assumed that the mixing mechanism of the mixer studied is similar to a diffusion process. This assumption was based on the work carried out in the field of batch mixing mentioned in chapter 2. It is not, however, easy to define what is meant by diffusive mixing nor is it easier to differentiate between it and the mixing produced by convection and shear. As a rough approximation we may assume that when the scale of turbulence causing the mixing is smaller than the sample size, the mechanism of mixing can be described as "diffusive".

If in a mixer a particle finds no concentration changes of its environment on its main path induced by the mixer motion, but only concentration gradients normal (perpendicular) to that main path, the mixing is caused by lateral turbulent deviations of the particles from their paths. Diffusive mixing occurs if the scale of lateral turbulence is smaller than the sample size.

In our rotary cylinder the materials are initially convectively mixed till the concentration contours (lines normal to the concentration gradients) coincide with the particle stream lines, then mixing becomes diffusive if the turbulence scale is small enough.

Quantitatively, a distinction between the contributions made by convection and diffusion in the mixing process is impossible and would also serve little value. The important thing is that after the mixing process has proceeded for some time, the diffusion rate in all probability becomes the limiting factor for the mixing rate. Furthermore, the quantitative expressions developed for diffusion processes can be extended well into the range of those transfer processes taking place by convection. Because of these reasons we feel justified in referring to the process by the term "diffusive mixing".

It might be argued, that the concentration alone as used in this study does not provide precise information about the mixture quality. This is not likely, however, because it is usually considered that in diffusive mixing the particles at a certain place in the mixture are completely and randomly mixed in a statistical sense irrespective of the local composition of the mixture. On the other hand, for a diffusion process there exists some proportionality between the rate at which the concentrations decrease their deviations from the average and the momentary values of these concentration deviations. The proportionality depends on the effectiveness of the mixing process to the extent that the ratio between the deviations from the average and the declining rate of these deviations is an adequate measure of the effectiveness of the mixing process, provided that the pattern of the deviations has become "self-preserving".

Although the sampling method adopted in our studies might not provide precise information about the mixture quality, it does still adequately provide the more important information about the rate of improvement of the mixture quality, because this rate could be considered to be identical with the rate of decrease of the concentration at any point in the mixture. In any case, we thought it unnecessary to adopt an alternative sampling procedure because apart from the practical design difficulties which had to be solved, the sampling method used did give fairly satisfactory results.

When the results of the measurements themselves are considered, it will be observed that the estimated mean values were far from satisfactory in the later parts of the curves. This is not, however, due to the sampling procedure itself but rather to the increase of the standard error of the mean as the sample proportions approach a value of $1:1$. This is a characteristic feature for sampling from a Binomial distribution since the value of the variance has a maximum when the proportion of the components is $1:1$. Ten samples were thus insufficient to give a high degree of accuracy.

The necessity to avoid mixing of the entering feed streams was of course determined by the objectives of the experimental investigation. Applying the mixer for mere mixing, previous mixing would of course be of advantage. However, for investigating the performance of the machine itself it was necessary that the deviations from the completely mixed state be made as large as was practically possible.

Thus the aim for introducing the modifications at the early part of the mixer was to delay the start of the investigated mixing action to a point immediately before the first sampling point. It was therefore reasonable to measure the mixer length from that point. Also because the decay in concentration difference was considered to be logarithmic with distance, we should be justified in measuring this length from any place within the range for which the consideration of logarithmic decay is valid. However taking some point in the modified part of the cylinder as zero point for distances would have been wrong because the mixing mechanism is different from the one we investigate and furthermore we had made no provisions for measuring the state of mixedness in this early part of the mixer.

It must be confessed, however, that the first sampling point located at 12.5 cm from the feed end of the mixer did not always give good results. This was especially true for the trials which had a high proportion of blue to red sand. By reference to figure 21, it will be observed for example that for the upper two trials, the composition of blue sand for this first sampling point is not plotted. The reason here was that the samples removed from this point contained more red particles than the following point. However the red particles were observed to be composed of scales, fines and dust. Accordingly it was concluded that since it was impossible to clean the sand perfectly, this debris tended to segregate in this part of the mixer and thus the samples removed from this first point had to be left out of consideration.

Leaving the previous considerations apart we shall now discuss the experimental results. From the four variables investigated it was found that only the relative proportions of the materials mixed had no effect on the rate of mixing. Although this was to be expected, since there is no reason to believe that a mixer can differentiate between particles of different colours, yet there seems to be no general agreement about this subject in batch mixing. For example

GAYLE and GARY (1960) from experimental studies came to the conclusion that proportion seems to have some effect on the mixing rate. On the other hand, LACEY (1954) from purely theoretical considerations came to the conclusion that mixing materials in different proportions only affects the early part of the process to which he referred as the transient part. According to him, once these transients disappear, the process will proceed at an equal rate for all proportions of materials mixed. The duration of the transient period is determined by the relative proportions of the materials, it is shortest for a 1 : 1 proportion and tends to become longer as the proportion of the materials mixed becomes wider. These conclusions are in good agreement with our results with the only difference that LACEY's plot of the diffusion equation does not show the same trends observed in our study. However, he plotted the diffusion equation in the form of the variance and not in the form of a difference in proportions, as we did, which fact can account for the disagreement between the two types of curves. It is clear, however, from our results that proportion has no effect on the mixing rate.

The other variables investigated are definitely related to one another and accordingly will be discussed together. We have observed that at a certain level of surface roughness, the mixing rate can be increased by increasing the rotational speed and decreasing the feed rate. On the other hand when the speed and feed rate are held constant, the rate of mixing can be increased by increasing the surface roughness. Taken together these observations indicate that potentially the degree of surface roughness of the rotating cylinder is the determining factor for the maximum mixing rates that can be achieved. Whether this maximum is achieved or not is determined by the speed and feed rate used under a certain set of operating conditions.

For any speed there seems to be no optimum feed rate at which the mixer should be operated in order to get the fastest degree of mixing. To acquire the fastest degree of mixing the mixer should be operated at a feed rate very close to that minimum value at which the used speed can still produce mixing. Also it is possible to acquire higher mixing rates from a combination of a low speed and a low feed rate than when a higher speed is used with a higher feed rate, irrespective of the total quantity of material accumulated in the mixer. These facts can be observed from table 8, in which the D values and the quantity of material accumulated for several combinations of speed and feed rate are given.

TABLE 8. Several D values for different operating conditions.

Speed (r.p.m.)	Feed rate (g/sec.)	D (cm²/sec.)	Mat. in mixer (g)
12	17.83	0.83	2850
14	22.17	0.73	3150
10	19.50	0.77	3850
14	22.17	0.73	3150
10	20.67	0.67	4190
12	22.00	0.68	3700

For a certain degree of surface roughness there is a limit to the improvements that can be brought about by increasing the rotational speed. To make

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the mass rotate at a higher speed the wall must exert a larger friction force on it. At the moment this needed friction force exceeds its maximum possible value, slipping takes place, causing the friction force to breakdown to a much lower value, whereupon the rotating wall loses its power to maintain the tumbling motion of the particles. The surface on which the tumbling of the particles takes place becomes incomplete, the quantity of material transported by the wall to the top of the accumulated heap of material becomes very small, and mixing takes place at a negligible rate. Also, because the friction force is related to the force pressing two sliding surfaces together, the breakdown in the friction force takes place at a lower speed for a low feed rate than for a high feed rate, irrespective of the effectiveness of the mixing performed in both cases.

The main conclusion that can be drawn from the discussion of the experimental results is that the performance of a rotating cylinder as a continuous mixing device for solids is entirely determined by complex friction phenomena, and that transverse mixing is the main mixing mechanism for such mixers.

CHAPTER 5

GENERAL DISCUSSION

Notwithstanding the obvious advantages of a continuous process, until now the industrial application of continuous mixing of solids is relatively limited. Batch mixing is still the usual method. This is probably due to two reasons. In the first place continuous mixing involves specific difficulties, which are not easily solved. In the second place there is a serious lack of knowledge about continuous mixing of solids. Up to the present this process has been scarcely studied.

There are several ways to carry out continuous mixing in practice. For instance a number of batch mixers might be arranged in series and the materials to be mixed are transferred in a continuous stream through them. This however does not seem to us to be an attractive method. Also one can use the intensive turbulence existing in pneumatic conveying systems or in fluidised beds. In both cases mixing takes place almost instantaneously. These methods however have several disadvantages. A "pneumatic mixer" has a very small hold-up; it is extremely sensitive to fluctuations in the feed streams; there is practically no buffering action. The emphasis will then be on a very good control of the feed streams. This disadvantage does not seem to be insurmountable. More serious however is the problem of separating the solids from the transporting air without segregation. In principle this is difficult to avoid, when the particles of the mixture differ in size and density, which in practice is usually the case. Fluidised beds give rise to the same objections, but to a somewhat different degree. This method has the additional disadvantage that it is difficult to fluidise a mixture of widely differing particles. The application of the mentioned methods will be very limited, unless further research reveals appropriate solutions for these difficulties.

More commonly used for continuous solids mixing are those mixers which produce flow and mixing by employing either a mechanical element rotating in a stationary trough or a rotating cylinder with or without internal flights and other lifting devices. The former are exemplified by the ribbon mixers

and the several possible adaptations of a screw conveyor to perform effective mixing, while the latter are represented by the horizontal or inclined rotary cylinders.

In this study two of the last types of mixers mentioned have been investigated. We have to emphasize, however, that our study is limited to the simplest case of mixing two free-flowing materials of particles of the same size, form and other properties, such as density, roughness, the tendency to agglomerate etc. It is clear that mixing, batch or continuous, will be much more difficult if the materials differ in these properties. Undoubtedly it is dangerous to generalize our results. In particular this holds true with respect to the observed mixing rates. It is questionable even whether good mixing, batch or continuous, is at all possible if the materials differ strongly. One might imagine that in the operating mixer a good degree of mixing is obtained, but this will be lost on stopping and unloading a batch mixer, and during the outflow and collection of the mixture produced by a continuous mixer, respectively. In our opinion a continuous mixer might be advantageous for hard mixing tasks. It is highly important that this will be investigated further.

On the other hand it is impossible to decide whether a mixture is good or bad, unless the purposes for which it is made are defined. This means that in some cases, a mixture which by certain standards would be considered bad, would still be acceptable for some practical purpose. Because mixing of dry solids is widely applied in industry, it is impossible to define requirements that satisfy all practical cases.

Although our study has been limited to a fundamentally simple case of mixing, it still has substantially improved our knowledge about the process. At the start of our work, quantitative nor qualitative information was available to us. Now we have a broader insight into the problem, we understand in broad lines what takes place in a continuous mixer and we know which factors play a more or less important role. Also our work provides good starting points for further investigations.

The mixers investigated differ strongly in the manner by which mixing is produced. One, a small scale "SPAANS" mixer, produces mixing by mechanical agitation, while the other a plain rotating cylinder, depends on gravitational and frictional forces. The difference between the two types of mixers bears some analogy with the difference between forced convection and natural convection combined with diffusion in heat and mass transfer operations. A common feature of the two mixers is that the completion of the mixing process is accelerated by an increase in rotational speed. At high speeds mixing becomes so rapid that only a small mixer would be required to perform the desired task. Actually the mixers have large capacities with respect to their sizes. Besides the speed also the feed rate has appreciable effect on the performance of the two mixers, although the effects of these two factors are more pronounced in the rotary cylinder than in the "SPAANS" mixer. For the rotary cylinder operating at a certain feed rate, a small change in r.p.m. can influence the mixing rate from rapid to slow or almost no mixing.

Both mixers show longitudinal mixing, but this phenomenon is much more pronounced in the "SPAANS" mixer than in the rotary cylinder. For the "SPAANS" mixer the length equivalent to a perfect mixer L', is increased by an increase of the mixer length L, increase of the pitches of screw and ribbon and by an increase of the rotational speed. Longitudinal mixing is effective in

smoothing out fluctuations in the composition of the feed streams and might even allow cruder methods of feeding the mixer. Clearly these different possibilities deserve further investigation.

It is very difficult to say how much the longitudinal mixing in the case of constant feed streams contributes to the improvement of the mixture quality. We think that in a rotary mixer longitudinal mixing is not of great importance. However, in a "SPAANS" type mixer it seems that longitudinal mixing contributes considerably to the total mixing because the concentration pattern is very irregular.

In the rotary cylinder transverse mixing is determined by friction phenomena while for the "SPAANS" mixer it seems to be related to the general design of the mixing element. In the latter, mixing takes place in a variable (nonconstant) way. Actually from the design of the mixing element one would expect that the improvement in mixture quality would take place by alternate steps of fast and slow mixing. Fast mixing would be expected at the meeting points of the slugs of material transported by the screw conveyor and the ribbon, while slow mixing would occur in the material as it slides along the helicoidal surfaces of the screw and the ribbon. Although there is not sufficient evidence to indicate that this actually happens in the mixer, such a description provides a reasonable explanation for the observed irregular behaviour of the mixing process.

Small changes in the design of the mixing element can bring about substantial improvement in the performance of the mixer. When the diameter of the screw conveyor was slightly under a certain limit value, the mixer did not function properly. Also when a mixing element with larger pitches of the screw conveyor and the ribbon was used, the distribution of the components of the mixture throughout the mixer's trough was better than when a mixing element with shorter pitches was used. Another point which might be overlooked is the simple fact that the performance of the "SPAANS" mixer is definitely influenced by the quality of the finish and the care taken in the construction of the mixing element. Special attention should be paid to the clearance made between the rotating ribbon and the stationary wall. This should be made as small as is practically possible in order to avoid undesirable logging of material in the bottom of the trough.

In view of the difference in mixing mechanisms the most appropriate way to study the two mixers differed. In all probability every type of continuous mixer would require a special experimental procedure and a special way of treating the results.

Because of the irregular character of the mixing process in the "SPAANS" mixer, the relation between the mixer length and a mixing index could not be expressed mathematically. For such a mixer the only characteristic for the mixing rate is the minimal length at which mixing is complete.

This is in contrast to the rotary cylinder. Here mixing is smooth and gradual. We attempted to study the mixing mechanism of the rotary cylinder by making an analogy with diffusion. An exact mathematical expression representing the process was difficult to acquire. Instead a simplified equation was used. Although such an equation allowed us to draw some useful conclusions, still it can only be considered as a very rough first approximation. Actually the used equation did not cover the results completely, nor is it expected to hold at all when materials with segregating tendencies are mixed. In this equation the volumetric flow rate was expressed as the feed rate divided by the bulk density of the

material at rest and was thus assumed to be constant from place to place in the mixer. This is true, but strictly speaking we were not completely justified in replacing the product $a^2 . v$. (see section 4.5) by the volumetric flow rate W/ρ, because the shape of the cross-section of the layer and so the ratio between the cross-sectional area and a^2 changed along the mixer. Allowance for the change of this ratio would entail rather complex mathematical equations, such as that reported by KRAMERS and CROOKEWIT (1952). The simple equation used, served however its purpose rather satisfactory, and as far as the present study is concerned a greater degree of accuracy was not considered to be essential.

Rotary cylinders (kilns) are often used not only for mixing but at the same time for handling a continuous flow of granular materials in drying, humidifying, heating, cooling, etc. For this reason they are still investigated by several workers. Examples of the work carried out in this field can be found in articles of VÀHL and KINGMA (1952) and KRAMERS and CROOKEWIT (1952). From theoretical considerations these authors developed equations for the volumetric flow, the hold-up, the mean residence time and the power requirements for plain horizontal and inclined cylinders. They also carried out some experimental work which shows that the results of their theoretical derivations are in good accordance with the values obtained from their experiments. Unfortunately this work came to our notice only after the experimental work carried out in this study was finished. No quantitative comparison can be made between our findings and theirs, since we did not determine most of the variables required for this purpose.

Their theoretical work was based on the assumption that no sliding takes place at the cylinder wall. They attribute longitudinal flow entirely to the axial component of the cascading movement of the particles. In our opinion this is only one of two limiting cases, the other limiting case being a movement entirely determined by sliding. Our results indicate that in practice both mechanisms operate simultaneously. The tangential friction force must be sufficient to result in the exceedance of the angle of repose by the accumulated material in order to maintain the cascading motion of the particles.

From dimensional analysis it might be expected, that the apparent diffusion coefficient in a rotary cylinder is proportional to the square of the diameter of the cylinder and to its rotational speed, provided that the other dimensions remain geometrically similar and the same materials are used. This would lead to the conclusion that the improvement in mixture quality for every increase in length, equal to one cylinder diameter, should be the same, for the same ratio between hold-up and cylinder volume. Our results do not agree with this; for a combination of low feed rate and low rotational speed the improvement of mixture quality per unit length of our rotary mixer was better than for a high feed rate and a high speed but the same hold-up. The only explanation is, that in the latter case, the contribution of sliding to the longitudinal transport is higher. This makes extrapolation of our results to other mixer dimensions rather risky. This is even more so for the case of the "SPAANS" mixer.

From our discussion it becomes evident that the performance of continuous solids mixers is not really determined by the mixing rate. Greater attention should be paid to their design with the object of increasing their effectiveness as solids mixing devices and to make them suitable for performing hard mixing tasks.

Similar conclusions have also been arrived at in batch mixing. A good example here is the work of RUMPF and MUELLER (1962). These authors studied the performance of centrifugal mixers and concluded that mixing time is not of decisive importance for the development and design of mixers for the handling of dry solids. More important factors are the design of the mixing elements, power consumption and material properties.

Thus length in continuous mixers and time in batch mixers should not be taken as the main criterions for the performance of solids mixers. Increasing the length or the time will not necessarily guarantee good results and in most cases would only be wasteful. For a continuous mixer, the length required for a certain job is determined by material properties, the characteristics of the feed streams and an economic balance between operating and capital costs.

<div align="center">CHAPTER 6</div>

SUMMARY

The continuous mixing of solids affords several advantages over batchwise operation. In view of the increasing importance of continuous mixing and the fact that up till now no or at least nearly no systematic experimental work has been carried out in this field, it was decided to start with such work. The results are reported in this thesis.

In chapter 2 the theoretical aspects of solids mixing are considered. The most important literature is reviewed. It appears that a rather extensive literature already exists about batch mixing; on the other hand there are only a few publications related to the theoretical aspects of continuous mixing. Consecutively the chapter deals with: the mechanisms of the process, the different methods of defining and analysing mixture quality (mixing index), the segregation effect, the rate of mixing, the differences between batch and continuous mixing and the residence time distribution in continuous flow systems.

From all this it appears that the physical properties of the materials play an important role; and only when the particles are uniform in size and other physical properties is it possible to describe and analyse the mixing process quantitatively. When however the materials differ in properties, the mixing process becomes extremely complex and is very difficult to handle quantitatively from both points of view of mixture quality as well as mixing rate. Furthermore it is concluded that the best method available to characterise a mixture of materials which shows no segregation is by means of the Chi-Square statistic. Also the conclusion is drawn that the ever present longitudinal mixing in continuous mixers can be studied by determination of the spread in residence times.

Since relatively little is known about continuous mixing, it was decided to start with a study of the simplest case, namely the mixing of uniform particles possessing the same properties. Accordingly the emphasis came to lie on machine characteristics. Therefore two common types of mixers were investigated.

In chapter 3 the experiments with a semi-technical "SPAANS" mixer are described. This mixer produces mixing by means of a screw conveyor surrounded by a helical ribbon which transports the material in a direction opposite to

that of the screw. The progress of the mixing process along the length of the machine was studied as a function of the rotational speed of the mixing element; for this purpose two different designs were used. The results were plotted as Chi-Square versus mixer length. It was observed that mixing proceeded rapidly but irregularly. The degree of mixing as function of length could not be expressed mathematically. The rate of mixing is only reflected by the minimal length necessary for apparent completion of mixing. This length is decreased for higher rotational speeds. The design of the mixing element appears to have a great influence on the performance of the mixer.

Next, the spread in residence times was measured; and this was observed to be appreciable. It was concluded that transverse mixing is more important than longitudinal mixing in order to achieve good results.

Chapter 4 reviews the experiments carried out with a plain, rotating, slightly inclined cylinder. The mixing mechanism in this machine is entirely different from that in the previously described mixer. Whereas in the "SPAANS" mixer a kind of "forced convection" is induced by mechanical means in the rotary cylinder something like "natural convection" in combination with "diffusion" takes place due to upward displacement of the material as a result of wall friction, followed by flow over the surface present at the angle of repose. The mixing in this cylinder proceeds gradually. In view of the mixing mechanism the experimental results for this case were treated in analogy with the smoothing out of concentration differences by diffusion. For this purpose $\Delta C / \Delta C_0$ was plotted versus mixer length. An attempt was made to state an exact equation for the mixing process; this proved to be impossible. Therefore a simple equation was used in order to compare the mixing rates observed under various operating conditions.

The influences of the rotational speed, the wall roughness of the cylinder, the feed rate and the proportion of the materials, on the mixing rate were investigated. Apparently the mixing rate is increased by increasing rotational speed, increasing wall roughness and decreasing feed rate. However, no optimal operating conditions were observed, although there are limits within which mixing proceeds properly. Proportion had no effect on the mixing rate.

Longitudinal mixing was studied for this mixer too; this proved to be very limited in extent, at least for the slight inclination used.

Finally the results are discussed in chapter 5, and in particular the mixing mechanisms are considered. The risks of generalising our results are emphasized. Some remarks about the practical application of continuous solids mixing are made.

ACKNOWLEDGEMENTS

help and advice on many problems, for accepting to be joint promotor, for his stimulating criticism and for helping with the correction of the manuscript.

I wish to express my sincere gratitude to Ir. W. A. BEVERLOO for his deep interest in the study, for the efforts he took to explain many problems and for all the help and time he freely gave throughout the work and during the preparation and correction of the manuscript.

I also wish to mention my special obligations for the help rendered, and I will always appreciate the good relations which I had with the staff of the laboratory. In particular I wish to thank Mr. H. TAP, Mr. G. VAN DEN BRINK and Mr. J. SACHTELEBEN for the excellent equipment they prepared, Miss I. TIMMER and Miss J. A. H. G. ALFERINK for typewriting a great part of the manuscript and Mr. K. WIGGERMAN for drawing the figures.

Special thanks are due to Professor Dr. H. A. LAUWERIER and Mr. J. F. FRANKENA of the Mathematical Centre in Amsterdam for their advice on the mathematics of the diffusion equation.

I owe special obligations to my wife for helping me with the analysis, the computational work and the typewriting of many parts of the manuscript. Many thanks are also due to my colleague Dr. L. H. A. HINDI for helping with some of the analytical work.

I am very grateful to all those who have contributed, in any way, to make this study possible.

<div align="center">CHAPTER 7</div>

SAMENVATTING

Het continu mengen van vaste stoffen biedt tegenover ladingsgewijs werken verschillende voordelen. In verband met de toenemende betekenis van continu mengen en de omstandigheid dat hierover tot nu toe geen of althans zeer weinig systematisch, experimenteel onderzoek is verricht, werd besloten een begin te maken met zulk onderzoek. De resultaten zijn vastgelegd in dit proefschrift.

In hoofdstuk 2 wordt een beschouwing gegeven over de theoretische aspecten van de menging van vaste stoffen. Daarbij wordt de voornaamste literatuur besproken. Het blijkt dat over ladingsgewijs mengen reeds een tamelijk omvangrijke literatuur bestaat; daarentegen zijn er slechts enkele publicaties die verband houden met de theoretische aspecten van continue menging. Achtereenvolgens worden in dit hoofdstuk besproken: de mechanismen van het proces, de verschillende methoden om de kwaliteit van een mengsel (graad van menging) te definiëren en te analyseren, het segregatie-effect, de snelheid van menging, de verschillen tussen ladingsgewijs en continu mengen en de spreiding in de verblijfsduur bij laatstgenoemde werkwijze.

Uit dit alles blijkt dat de fysische eigenschappen van de te mengen materialen een belangrijke rol spelen; slechts wanneer de te mengen deeltjes uniform zijn in afmetingen en fysische eigenschappen is het mogelijk het mengproces in kwantitatieve zin te beschrijven en te analyseren. Wanneer de materialen echter verschillende eigenschappen hebben wordt het mengproces uitermate complex en zeer moeilijk kwantitatief te behandelen, zowel ten aanzien van de kwaliteit van het mengsel als van de mengsnelheid. Voorts wordt geconcludeerd, dat de beste methode om een mengsel van materialen, die geen segregatie

vertonen, te karakteriseren is, het gebruik van de Chi-kwadraat statistiek. Ook wordt de conclusie getrokken dat de in een continu menger steeds optredende longitudinale menging goed kan worden bestudeerd door de spreiding in de verblijfsduur van de materialen te bepalen.

Aangezien over continue menging nog zo weinig bekend is, werd besloten te beginnen met een studie van het eenvoudigste geval, nl. van menging van uniforme deeltjes met dezelfde eigenschappen. Het accent van het onderzoek kwam daardoor te liggen op de eigenschappen van de mengapparatuur.

Daartoe werden twee gebruikelijke typen van mengers in het onderzoek betrokken.

In hoofdstuk 3 worden de experimenten met een semitechnische "Spaans" menger beschreven. Deze brengt menging teweeg langs mechanische weg met behulp van een transportschroef waar omheen zich een schroeflint bevindt, dat het materiaal verplaatst in een richting tegengesteld aan de transportrichting van de schroef. In deze menger werd het verloop van de menging in de lengterichting van het apparaat bestudeerd als functie van het aantal omwentelingen van de combinatie schroef/lint; daarbij werden twee verschillende constructies gebruikt. De resultaten werden weergegeven in de vorm van grafieken met Chi-kwadraat als functie van de lengte van het apparaat. Geconstateerd werd dat de menging snel plaats vindt, doch onregelmatig verloopt. De graad van menging als functie van de lengte kon niet in een vergelijking worden uitgedrukt. De mengsnelheid komt alleen tot uitdrukking in de lengte die benodigd is om een volledige menging te bereiken. Hogere toerentallen verkorten de benodigde lengte. Het ontwerp van de combinatie schroef/lint bleek een grote invloed te hebben op de werking.

Voorts werd de spreiding in de verblijfsduur gemeten; in deze menger bleek de spreiding aanzienlijk te zijn. Geconcludeerd werd dat voor het verkrijgen van een goed resultaat de menging loodrecht op de transportrichting belangrijker is dan de longitudinale menging.

Hoofdstuk 4 geeft een overzicht van experimenten verricht met een eenvoudige roterende, enigszins hellend opgestelde, cylinder. Het mechanisme van menging in dit apparaat is geheel verschillend van dat in de eerder beschreven menger. Terwijl in de "Spaans"-menger een soort van "gedwongen convectie" tot stand wordt gebracht langs mechanische weg, treedt in de roterende cylinder meer een "natuurlijke convectie" in combinatie met "diffusie" op, veroorzaakt door mee omhoog nemen van het materiaal tengevolge van wrijving langs de wand gevolgd door stroming over het onder de storthoek staande oppervlak. De menging geschiedt in deze cylinder zeer geleidelijk. In verband met het mechanisme werden de onderzoekresultaten in dit geval verwerkt in analogie met concentratievereffening door diffusie. Daartoe werden grafieken gemaakt met $\Delta C/\Delta C_0$ als functie van de lengte van de menger. Getracht werd een nauwkeurige vergelijking voor het verloop van de menging op te stellen; dit bleek niet mogelijk. Daarom werd van een eenvoudige vergelijking gebruik gemaakt om de mengsnelheid onder verschillende bedrijfsomstandigheden te vergelijken.

Onderzocht werd de invloed van het aantal omwentelingen, de ruwheid van de wand van de cylinder, de toevoersnelheid en de verhouding tussen de hoeveelheden materialen. De mengsnelheid bleek groter te worden door verhoging van het toerental, vergroting van de wandruwheid en vermindering van de toevoersnelheid. Echter werden geen optimale bedrijfsomstandigheden

waargenomen, wel zijn er grenzen waarbinnen de menging goed verloopt. Voorts werd geen invloed van de mengverhouding van de materialen gevonden.

Ook bij deze menger werd de longitudinale menging bestudeerd; deze bleek, althans bij de onderzochte hellingshoek, zeer beperkt te zijn.

Ten slotte worden de resultaten in hoofdstuk 5 in beschouwing genomen, waarbij in het bijzonder op de mechanismen van de menging wordt ingegaan. Gewaarschuwd wordt tegen generaliseren van de resultaten. Enkele opmerkingen over de praktische uitvoering van de continuë menging van vaste stoffen worden gemaakt.

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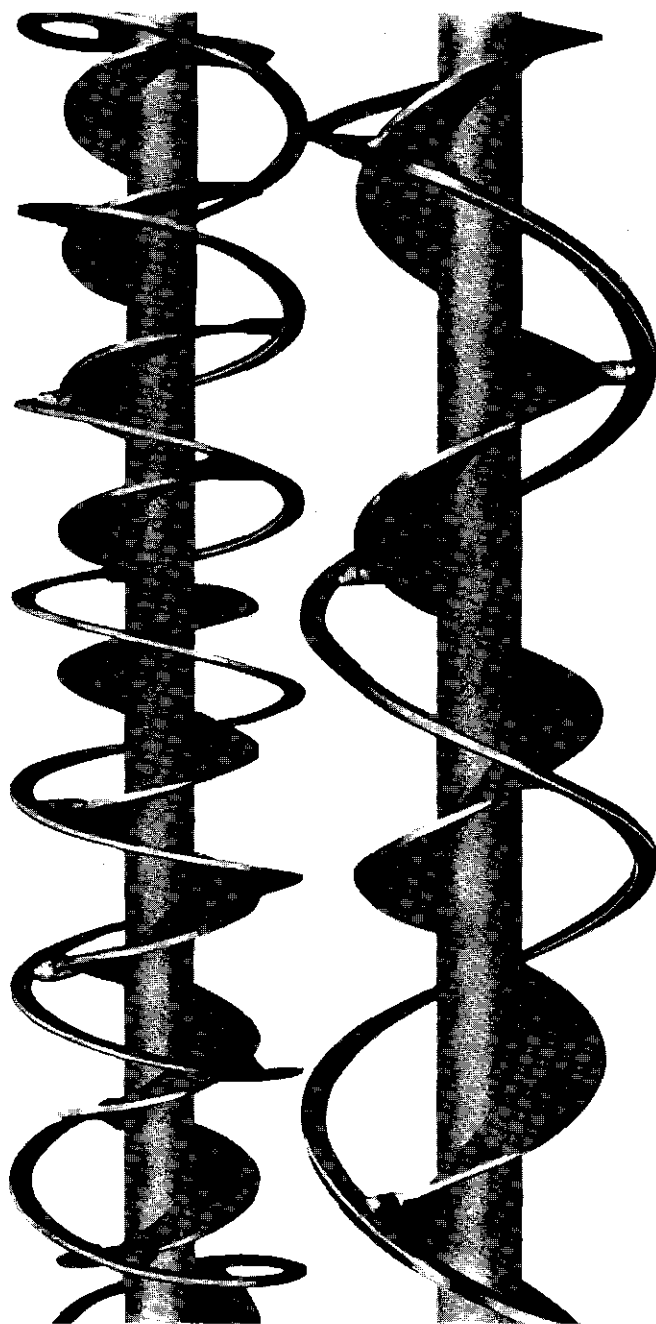
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PLATE 1. The two types of mixing element used in the experiments. The difference between the pitch lengths can be clearly seen.

PLATE 2. Part of the "SPAANS" mixer and the feed assembly. It can be seen that most of the material lies on one side of the axis of the mixing element and acquires a wavy surface. The sampling guide is also seen in its sampling position.
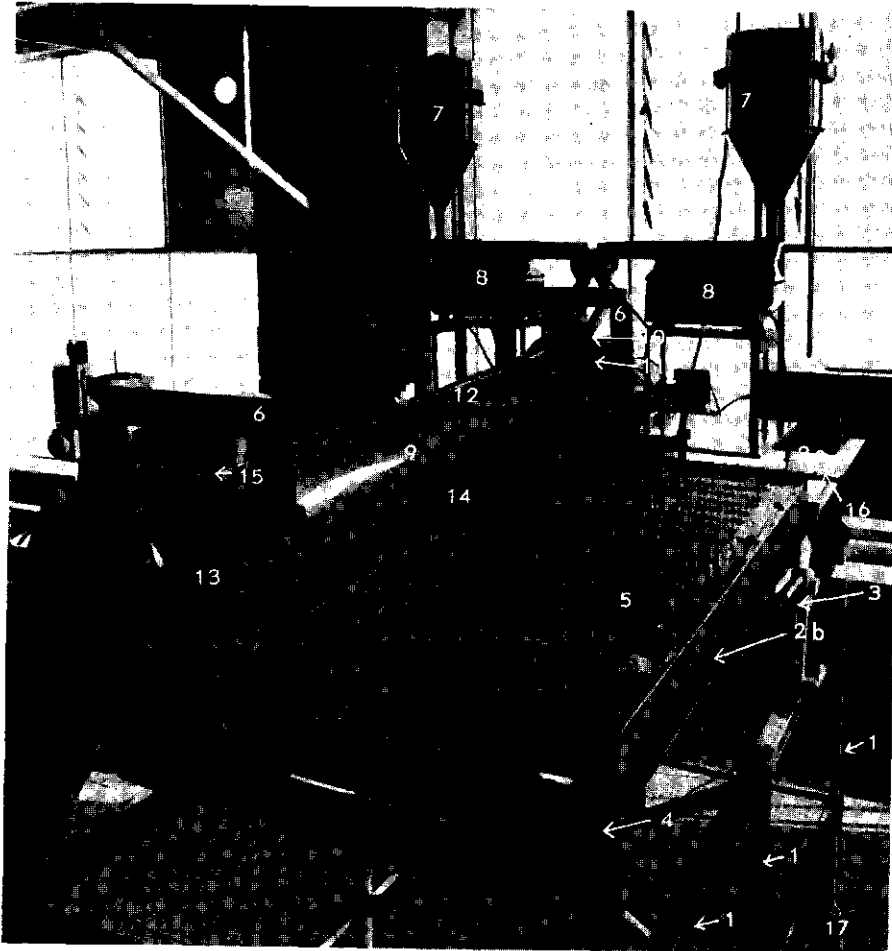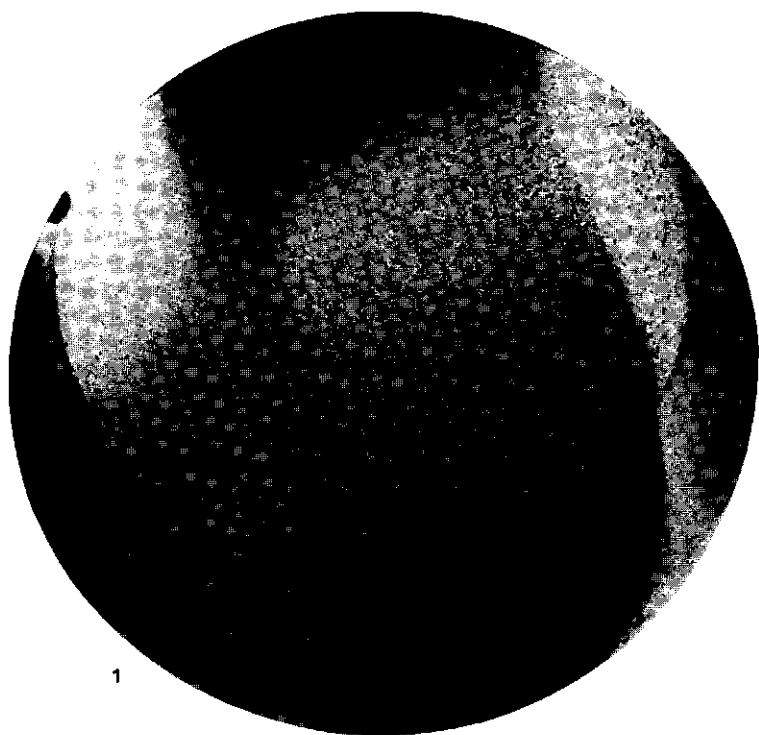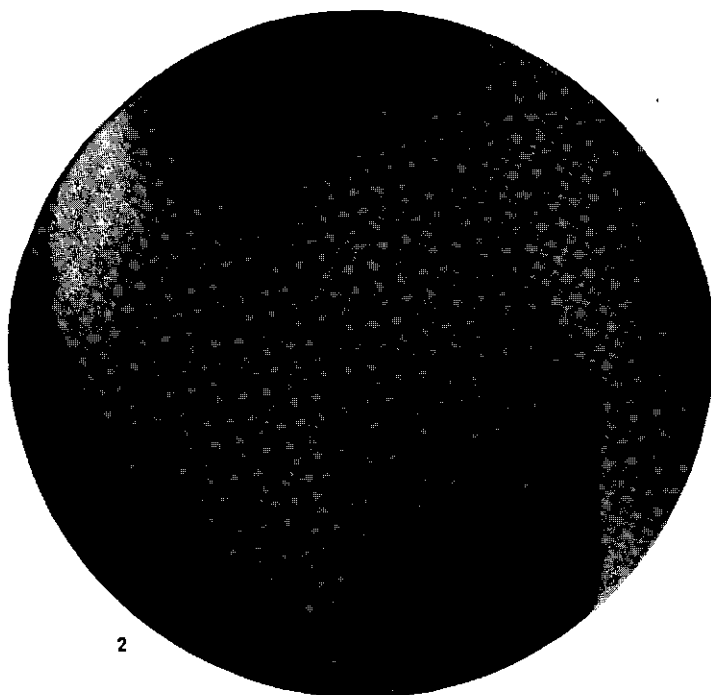
PLATE 3. Experimental rotary cylinder mixer. The indicated numbers refer to the different parts described in the text.

PLATE 4. Upper picture shows the normal cascading surface of the particles. Lower picture shows surface of the accumulated layer when the operating conditions of the mixer prevent the cascading motion of the particles from taking place.