and reliability have been the result of this effort, which has been
driven by the increased availability of special purpose computers
and the data processing capability of low-cost personal computers.
Table 21–21 lists those commonly used and gives their sensitiv-
ity. As a result of this, custom-designed weighing equipment has
become an important alternative to standardized or “off the shelf”
designs. This is especially true when there is a necessity to modify
a standard design, which often is more expensive than a custom
design.

SOLIDS MIXING

PrINCIPLEs OF SolIDS MIXING

Industrial Relevance of Solids Mixing The mixing of powders,
particles, flakes, and granules has gained substantial economic impor-
tance in a broad range of industries, including, e.g., the mixing of
human and animal foodstuff, pharmaceutical products, detergents,
chemicals, and plastics. As in most cases the mixing process adds sig-
nificant value to the product, the process can be regarded as a key unit
operation to the overall process stream.

By far the most important use of mixing is the production of a
homogeneous blend of several ingredients which neutralizes vari-
ations in concentration. But if the volume of material consists of one
ingredient or compound exhibiting fluctuating properties caused by
an upstream production process, or inherent to the raw material itself,
the term homogenization is used for the neutralization of these fluctu-
ations. By mixing, a new product or intermediate is created for which
the quality and price are very often dependent upon the efficiency of
the mixing process. This efficiency is determined both by the materi-
als to be mixed, e.g., particle size and particle-size distribution, den-
sity, and surface roughness, and by the process and equipment used
for performing the mixing. The design and operation of the mixing
unit itself have a strong influence on the quality produced, but
upstream material handling process steps such as feeding, sifting,
weighing, and transport determine also both the quality and the
capacity of the mixing process. Downstream processing may also
destroy the product quality due to segregation (demixing). Continu-
ous mixing is one solution which limits segregation by avoiding storage
equipment.

The technical process of mixing is performed by a multitude of
equipment available on the market. However, mixing processes are
not always designed with the appropriate care. This causes a signifi-
cant financial loss, which arises in two ways:

1. The quality of the mix is poor: In cases where the mixing pro-
duces the end product, this will be noticed immediately at the prod-
uct’s quality inspection. Frequently, however, mixing is only one in a
series of further processing stages. In this case, the effects of unsatis-
factory blending are less apparent, and might possibly be overlooked
by the detriment of final product quality.

2. The homogeneity is satisfactory but the effort employed is too
great (overmixing). Overmixing in batch blending is induced by an over-
long mixing time or too long a residence time in the case of continuous
blending. This leads to increased strain on the mixture which can have an
adverse effect on the quality of sensitive products. Furthermore, larger
or more numerous pieces of equipment must be than would be
necessary in the case of an optimally configured mixing process.

Mixing Mechanisms: Dispersive and Convective Mixing

The mixing process can be observed in diagrammatic form as an over-

<table>
<thead>
<tr>
<th>TABLE 21-21 Weight Sensing Devices and Sensitivity</th>
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<tbody>
<tr>
<td>Device</td>
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<tr>
<td>--------</td>
</tr>
<tr>
<td>Beam-Microwave</td>
</tr>
<tr>
<td>Beam-Differential Transformer</td>
</tr>
<tr>
<td>Strain Gauge Type Load Cell</td>
</tr>
<tr>
<td>Magnetic Force Restoration Transducer</td>
</tr>
<tr>
<td>Variable Capacitance Transducer</td>
</tr>
</tbody>
</table>

Data courtesy of Kg Systems, Inc., Bloomsfield, NJ.

FIG. 21-77 The mixing process can be observed in diagrammatic form as an
overlap of dispersion and convection (Fig. 21-77). Movement of the par-
ticulate materials is a prerequisite of both mechanisms. Dispersion is
understood to mean the completely random change of place of the
individual particles. The frequency with which the particles of ingre-
dient A change place with those of another is related to the number of
particles of the other ingredients in the direct vicinity of the particles
of ingredient A. Dispersion is therefore a local effect (micromixing)
taking place in the case of premix systems where a number of particles of
different ingredients are in proximity, leading to a fine mix localized
to very small areas. If the ingredients are spatially separated at the
beginning of the process, long times will be required to mix them
through dispersion alone, since there is a very low number of assorted
neighbors. Dispersion corresponds to diffusion in liquid mixtures.
However, in contrast to diffusion, mixing in the case of dispersion is
not caused by any concentration gradient. The particles have to be in
motion to get dispersed. Convection causes a movement of large
groups of particles relative to each other (macromixing). The whole
volume of material is continuously divided up and then mixed again
after the portions have changed places (Fig 21-77). This forced con-
vection can be achieved by rotating elements. The dimension of the
groups, which are composed of just one unmixed ingredient, is con-
tinuously reduced splitting action of the rotating paddles. Convection
increases the number of assorted neighbors and thereby promotes
the exchange processes of dispersive mixing. A material mass is divided up

![Diagram of Mixing by Dispersion and Convection](image-url)
or convectively mixed through the rearrangement of a solid’s layers by rotating devices in the mixer or by the fall of a stream of material in a static gravity mixer, as discussed below.

Segregation in Solids and Demixing If the ingredients in a solids mixture possess a selective, individual motional behavior, the mixture’s quality can be reduced as a result of segregation. As yet only a partial understanding of such behavior exists, with particle movement behavior being influenced by particle properties such as size, shape, density, surface roughness, forces of attraction, and friction. In additional, industrial mixers each possess their own specific size, shape, density, surface roughness, forces of attraction, and friction behavior being influenced by particle properties such as only a partial understanding of such behavior exists, with particle mixture’s quality can be reduced as a result of segregation. As yet solids mixture possess a selective, individual motional behavior, the static gravity mixer, as discussed below.

Agglomeration segregation occurs through the preferential self-agglomeration of one component in a two-ingredient mixture (Fig. 21-78a). Agglomerates form when there are strong interparticle forces, and for these forces to have an effect, the particles must be brought into close contact. In the case of agglomerates, the particles stick to one another as a result, e.g., of liquid bridges formed in solids, if a small quantity of moisture or other fluid is present. Electrostatic and van der Waals forces likewise induce cohesion of agglomerates. Van der Waals forces, reciprocal induced and dipolar, operate particularly upon finer grains smaller than 30 μm and bind them together. High-speed impellers or knives are utilized in the mixing chamber to create shear forces during mixing to break up these agglomerates. Agglomeration can, however, have a positive effect on mixing. If a solids mix contains a very fine ingredient with particles in the submicrometer range (e.g., pigments), these fine particles coat the coarser ones. An ordered mixture occurs which is stabilized by the van der Waals forces and is thereby protected from segregation.

Floation segregation can occur if a solids mix is vibrated, where the coarser particles float up against the gravity force and collect near the top surface, as illustrated in Fig. 21-78b for the case of a large particle in a mix of finer material. During vibration, smaller particles flow into the vacant space created underneath the large particle, preventing the large particle from reclaiming its original position. If the large particle has a higher density than the fines, it will compact the fines, further reducing their mobility and the ability of a large particle to pass through the fines. If the effect of the larger particle’s geometry there is little probability that this effect will run in reverse and that a bigger particle will take over the place left by a smaller one which has been lifted up. The large particle in this case would also have to displace several smaller ones. As a result the probability is higher that coarse particles will climb upward with vibration.

Percolation segregation is by far the most important segregation effect, which occurs when finer particles trickle down through the gaps between the larger ones (Fig. 21-78c). These gaps act as a sieve. If a solids mixture is moved, gaps briefly open up between the grains, allowing finer particles to selectively pass through the particle bed. Granted a single layer has a low degree of separation, but a bed of powder consists of many layers and interconnecting grades of particles which taken together can produce a significant division between fine and coarse grains (see Fig. 21-78), resulting in widespread segregation. Furthermore, percolation occurs even where there is but a small difference in the size of the particles (250- and 300-μm particles) [J. C. Williams, Fuel Soc. J., University of Sheffield, 14, 29 (1963)]. The most significant economical example is the poured heap appearing when filling and discharging bunkers or silos. A mobile layer with a high-speed gradient forms on the surface of such a cone, which, like a sieve, bars larger particles from passing into the cone’s core. Large grains on the cone’s mantle obviously slide or roll downward. But large, poorly mixed areas occur even inside the cone. Thus filling a silo or emptying it from a central discharge point is particularly critical. Revealing of such segregated heaps can be achieved through mass flow discharge; i.e., the silo’s contents move downward in blocks, slipping at the walls, rather than emptying from the central core (funnel flow). (See Sec. 19, “Solids Handling.”)

Transport Segregation This encompasses several effects which share the common factor of a gas contributing to the segregation processes. Trajectory and fluidized segregation can be defined, first, as occurring in cyclones or conveying into a silo where the particles are following the individual trajectories and, second, in fluidization. During fluidization particles are exposed to drag and gravity forces which may lead to a segregation.

FIG. 21-78 Four mechanisms of segregation, following Williams.
mixing has to be defined. Here one has to specify what property characterizes a mixture, examples being composition, particle size, and temperature. The end goal of a mixing process is the uniformity of this property throughout the volume of material in the mixer. There are circumstances in which a good mix requires uniformity of several properties, e.g., particle size and composition. The mixture’s condition is traditionally checked by taking a number of samples, after which these samples are examined for uniformity of the property of interest. The quantity of material sampled or sample size and the location of these samples are essential elements in evaluating a solids mixture.

Sample size thus represents the resolution by which a mixture can be judged. The smaller the size of the sample, the more closely the mixture’s condition in the mixture is scrutinized (P. V. Dankwerts, The Definition and Measurement of Some Characteristics of Mixtures, Appl. Sci. Res., 27(1952). Specifying the size of the sample is therefore an essential step in analyzing a mixture’s quality, since it quantifies the mixing task from the outset. The size of the sample can only be meaningfully specified in connection with the mixture’s further application. In pharmaceutical production, active ingredients must be equally distributed; e.g., within the individual tablets in a production batch, the sample size for testing the condition of a mixture is one tablet. In less critical industries the sample size can be in tons. The traditional and general procedure is to take identically sized samples of the mixture from various points at random and to analyze them in an off-line analysis. Multielement mixtures can also be described as twin ingredient mixes when a particularly important ingredient, e.g., the active agent in pharmaceutical products, is viewed as a tracer element and all the other constituents are combined into one common ingredient. This is a simplification of the statistical description of solids mixtures. When two-element mixtures are being examined, it is sufficient to trace the concentration path of just one ingredient, the tracer. There will be a complementary concentration of the other ingredients. The description is completely analogous when the property or characteristic feature in which we are interested is not the concentration but is, e.g., moisture, temperature, or the particle’s shape. If the tracer’s concentration in the mixture is $p$ and that of the other ingredients is $q$, we have the following relationship: $p + q = 1$. If you take samples of a specified size from the mixture and analyze them for their content of the tracer, the concentration of tracer $x_i$ in the samples will fluctuate randomly around that tracer’s concentration $p$ in the whole mixture (the “base whole”). Therefore a mixture’s quality can only be described by using statistical means. The smaller the fluctuations in the sample’s concentration $x_i$ around the mixture’s concentration $p$, the better its quality. This can be quantified by the statistical variance of sample concentration $\sigma^2$, which consequently is frequently defined as the degree of mixing.

There are many more definitions of mix quality in literature on the subject, but in most instances these relate to an initial or final variance and are frequently too complicated for industrial application (K. Sommer, Mixing of Solids, in Ullmann’s Encyclopaedia of Industrial Chemistry, vol. B4, Chap. 27, VCH Publishers Inc., 1992). The theoretical variance for finite sample numbers is calculated as follows:

$$\sigma^2 = \frac{1}{N^2} \sum_{i=1}^{N} (x_i - p)^2$$

(21-58)

The relative standard deviation RSD is used as well for judging mixture quality. It is defined by

$$RSD = \sqrt{\frac{\sigma^2}{p}}$$

(21-59)

The variance is obtained by dividing up the whole mix, the base whole, into $N$ samples of the same size and determining the concentration $x_i$ in each sample. Figure 21-79 illustrates that smaller samples will cause a larger variance or degree of mixing.

If one analyzes not the whole mix but a number $n$ of randomly distributed samples across the base whole, one determines instead the sample variance $S^2$. If this procedure is repeated several times, a new value for the sample variance will be produced on each occasion, resulting in a statistical distribution of the sample variance. Thus each $S^2$ represents an estimated value for the unknown variance $\sigma^2$. In many cases the concentration $p$ is likewise unknown, and the random sample variance is then defined by using the arithmetical average $\mu$ of the sample’s concentration $x_i$.

$$S^2 = \frac{1}{n-1} \sum_{i=1}^{n} (x_i - \mu)^2$$

(21-60)

Random sample variance data are of little utility without knowing how accurately they describe the unknown, true variance $\sigma^2$. The variance is therefore best stated as a desired confidence interval for $\sigma^2$. The confidence interval used in mixing is mostly a unilateral one, derived by the $\chi^2$ distribution. Interest is focused on the upper confidence limit, which, with a given degree of probability, will not be exceeded by the variance [Eq. (21-61)] (J. Raasch and K. Sommer, The application of statistical test procedures in the field of mixing technology, in German, Chemical Engineering, 62(1), 17–22 (1990)) which is given by

$$W\left(\sigma^2 < (n - 1) \frac{S^2}{\chi^2}\right) = 1 - \Phi(\chi^2)$$

(21-61)

Figure 21-80 illustrates how the size of the confidence interval normalized with the sample variance decreases as the number of random samples $n$ increases. The confidence interval depicts the accuracy of the analysis. The smaller the interval, the more exactly the mix quality can be estimated from the measured sample variance. If there are few samples, the mix quality’s confidence interval is very large. An evaluation of the mix quality with a high degree of accuracy (a small confidence interval) requires that a large number of samples be taken and analyzed, which can be expensive and can require great effort. Accuracy and cost of analysis must therefore be balanced for the process at hand.

Example 3: Calculating Mixture Quality Three tons of a sand (80 percent by weight) and cement (20 percent by weight) mix has been produced. The quality of this mix has to be checked. Thirty samples at 2 kg of the material mixture have been taken at random, and the sand content in these samples established.
The mass fraction of the sand $x_{\text{sand}}$ in the samples comes to

$$\bar{x} = \frac{1}{n} \sum_{i=1}^{n} x_i = \frac{1}{30} \sum_{i=1}^{30} x_i = 0.797$$

The degree of mixing defined as the variance of the mass fraction of sand in the mix needs to be determined. It has to be compared with the variance for a fully segregated system and the ideal variance of a random mix. First, the random sample variance $S^2$ [Eq. (21-59)] is calculated, and with it an upper limit for the true variance $\sigma^2$ can then be laid down. The sand’s average concentration $\mu$ in the whole 3-ton mix is estimated by using the random sample average $\bar{x}$:

$$\sigma^2 = \frac{1}{n-1} \sum_{i=1}^{n} (x_i - \mu)^2 = \frac{1}{29} \sum_{i=1}^{30} (x_i - \mu)^2$$

$$= \frac{1}{29} \left(3 \times 0.047^2 + 7 \times 0.027^2 + 5 \times 0.007^2 + 6 \times 0.013^2 + 7 \times 0.033^2 + 2 \times 0.053^2\right)$$

$$= 9.04 \times 10^{-4}$$

Ninety-five percent is set as the probability $W$ determining the size of the confidence interval for the variance $\sigma^2$. An upper limit (unilateral confidence interval) is then calculated for variance $\sigma^2$:

$$W\left[\sigma^2 < \frac{(n-1) S^2}{\chi^2_{0.05}}\right] = 0.95 = 1 - \Phi(\chi^2_{0.05}) = 0.95$$

From the table of the $\chi^2$ distribution summation function (in statistical teaching books) $\Phi(\chi^2, n-1)$ the value 17.7 is derived for 29 degrees of freedom. Figure 21-80 allows a fast judgment of these values without consulting statistical tables. Values for $(n-1)\chi^2_{0.05}$ are shown for different number of samples $n$.

$$\sigma^2 < \frac{(n-1) S^2}{\chi^2_{0.05}} = 29 \times \frac{9.04 \times 10^{-4}}{17.7} = 14.8 \times 10^{-4}$$

It can therefore be conclusively stated with a probability of 95 percent that the mix quality $\sigma^2$ is better (equals less) than $14.8 \times 10^{-4}$.

**Ideal Mixtures** A perfect mixture exists when the concentration at any randomly selected point in the mix in a sample of any size is the same as that of the overall concentration. The variance of a perfect mixture has a value of 0. This is only possible with gases and liquids which can be mixed molecularly and where sample volumes of the mixture are many times larger than its ingredients, i.e., molecules. In the case of solids mixtures, particle size must be considered in comparison to both sample size and sensor area. Thus $\sigma^2$ depends on the size of the sample (Fig. 21-81). There are two limiting conditions of maximum homogeneity which are the equivalent of a minimum variance: an ordered and a random mixture.

**Ordered Mixtures** The components align themselves according to a defined pattern. Whether this ever happens in practice is debatable. There exists the notion that because of interparticle processes of attraction, this mix condition can be achieved. The interparticle forces find themselves in an interplay with those of gravity and other dispersive forces, which would prevent this type of ordered mix in the case of coarser particles. Interparticle forces predominate in the case of finer particles, i.e., cohesive powders. Ordered agglomerates or layered particles can arise. Sometimes not only the mix condition but also the mixing of powders in which these forces of attraction are significant is termed ordered mixing [H. Egermann and N. A. Orr, Comments on the paper “Recent Developments in Solids Mixing” by L. T. Fan, Y. Chen, and F. S. Lai, Powder Technology, 68, 195–196 (1991)]. However, Egermann [L. T. Fan, Y. Chen, and F. S. Lai, Recent Developments in Solids Mixing, Powder Technology, 61, 255–287 (1990)] points to the fact that one should only use ordered mixing to describe the condition and not the mixing of fine particles using powerful interparticle forces.

**Random Mixtures** A random mixture also represents an ideal condition. It is defined as follows: A uniform random mix occurs when the probability of coming across an ingredient of the mix in any subsection of the area being examined is equal to that of any other point in time for all subsections of the same size, provided...
that the condition exists that the particles can move freely. The variance of a random mixture is calculated as follows for a two-component blend in which the particles are of the same size [P. M. C. Lacey, The Mixing of Solid Particles, Trans. Instn. Chem. Engrs., 21, 53–59 (1943)]:

\[ \sigma^2 = \frac{p \cdot q}{n_p} \]  
(21-63)

where \( p \) is the concentration of one of the ingredients in the mix, \( q \) is the other \( (q = 1 - p) \), and \( n_p \) is the number of particles in the sample. Note that the variance of the random mix grows if the sample size decreases. The variance for a completely segregated system is given by:

\[ \sigma_{\text{segregated}}^2 = p \cdot q \]  
(21-64)

Equation (21-63) is a highly simplified model, for no actual mixture consists of particles of the same size. It is likewise a practical disadvantage that the number of particles in the sample has to be known in order to calculate variance, rather than the usually specified sample volume. Stange calculated the variance of a random mix in which the ingredients possess a distribution of particle sizes. His approach is based on the fact that an ingredient possessing a distribution in particle size by necessity also has a distribution in particle mass. He made an allowance for the average mass \( m_p \) and \( m_q \) of the particles in each component and the particle mass’s standard deviation \( \sigma_p \) and \( \sigma_q \) [K. Stange, Die Mischgüte einer Zufallsmischung als Grundlage zur Beurteilung von Mischversuchen (The mix quality of a random mix as the basis for evaluating mixing trials), Chem. Eng., 26(6), 331–337 (1954)]. He designated the variability \( c \) as the quotient of the standard deviation and average particle mass, or

\[ c_p = \frac{\sigma_p}{m_p}, \quad c_q = \frac{\sigma_q}{m_q} \]  
(21-65)

Variability is a measure for the width of the particle-size distribution. The higher the value of \( c \), the broader the particle-size distribution.

The size of the sample is now specified in practice by its mass \( M \) and no longer by the number of particles \( n_p \) as shown in Eq. (21-63). The variance in random mixture for the case of two-component mixes can be given by

\[ \sigma^2 = \frac{p \cdot q}{M} \left[ m_p (1 + c_p^2) + m_q (1 + c_q^2) \right] \]  
(21-66)

Equation (21-65) estimates the variance of a random mixture, even if the components have different particle-size distributions. If the components have a small size (i.e., small mean particle mass) or a narrow particle-size distribution, that is, \( c_p \) and \( c_q \) are low, the random mix’s variance falls. Sommer has presented mathematical models for calculating the variance of random mixtures for particulate systems with a particle-size distribution (Karl Sommer, Sampling of Powders and Bulk Materials, Springer-Verlag Berlin, 1986, p. 164). This model has been used for deriving Fig. 21-81.

Measuring the Degree of Mixing The mixing process uniformly distributes one or more properties within a quantity of material. These can be physically recordable properties such as size, shape, moisture, temperature, or color. Frequently, however, it is the mixing of chemically differing components which forms the subject under examination. Off-line and on-line procedures are used for this examination (compare to subsection “Particle-Size Analysis”). Off-line procedure: A specified portion is (randomly or systematically) taken from the volume of material. These samples are often too large for a subsequent analysis and must then be split. Many analytical processes, e.g., the chemical analysis of solids using infrared spectroscopy, require the samples to be prepared beforehand. At all these stages there exists the danger that the mix status within the samples will be changed. As a consequence, when examining a mixing process whose efficiency can be characterized by the variance expression \( \sigma_{\text{process}}^2 \), all off- and on-line procedures give this variance only indirectly:

\[ \sigma_{\text{observed}}^2 = \sigma_{\text{process}}^2 + \sigma_{\text{measurement}}^2 \]  
(21-67)

The observed variance \( \sigma_{\text{observed}} \) also contains the variance \( \sigma_{\text{measurement}} \) resulting from the test procedure and which arises out of errors in the systematic or random taking, splitting, and preparation of the samples and from the actual analysis. A lot of attention is often paid to the accuracy of an analyzer when it is being bought. However, the preceding steps of sampling and preparation also have to fulfill exacting requirements so that the following can apply:

\[ \sigma_{\text{process}}^2 \gg \sigma_{\text{measurement}}^2 \Rightarrow \sigma_{\text{process}}^2 = \sigma_{\text{observed}}^2 \]  
(21-68)

Figure 21-82 illustrates the impact of precision of the determination of mixing time for batch mixers. It is not yet possible to theoretically forecast mixing times for solids, and therefore these have to be ascertained by experiments. The traditional method of determining mixing times is once again sampling followed by off-line analysis. The mixer is loaded and started. After the mixer has been loaded with the ingredients in accordance with a defined procedure, it is run and samples are taken from it at set time intervals. To do this the mixer usually has to be halted. The concentration of the tracer in the samples is established, and the random sample variance \( S^2 \) ascertained. This random sample variance serves as an estimated value for the variance \( \sigma_{p}^2 \) which defines the mixture’s condition. All analyses are burdened by errors, and this is expressed in a variance \( \sigma_{p}^2 \) derived from the sampling itself and from the analysis procedure. Initially there is a sharp fall in the random sample variance, and it runs asymptotically toward a final value of \( \sigma_{p}^2 \) as the mixing time increases. This stationary end value \( \sigma_{p}^2 \) is set by the variance of the mix in the stationary condition \( \sigma_2^2 \), for which the minimum would be the variance of an ideal random mix, and the variance \( \sigma_2^2 \) caused by errors in the analyzing process. The mixing time denotes that period in which the experimental random sample variance \( S^2 \) falls within the confidence interval of the stationary final condition \( \sigma_2^2 \). Two cases can be considered. In the first case with large measurement errors, \( \sigma_2^2 \) is determined by the analyzing process itself since for sufficient mixing time the mixing process’s fluctuations in its
stationary condition are much smaller than those arising out of the analysis or $\sigma_a < \sigma_b$. In this case, the mixing process can only be tracked at its commencement, where $\sigma_a > \sigma_b$. The "mixing time" $t_m$ obtained under these conditions does not characterize the process. In the second case where the measurement errors are small, or $\sigma_a > \sigma_b$, the analyzing process is sufficiently accurate for the mixing process to be followed through to its stationary condition. This allows an accurate determination of the true mixing time $t^*$. The "mixing time" $t_m$ obtained on the basis of an unsatisfactory analysis is always deceptively shorter than the true time $t^*$.

**On-line Procedures** Advances in sensor technology and data processing are enabling an increased number of procedures to be completely monitored using on-line procedures. The great leap forward from off-line to on-line procedures lies in the fact that the whole process of preparing and analyzing samples has been automated. As a result of this automation, the amount of collectible test data has risen considerably, thereby enabling a more comprehensive statistical analysis and, in ideal cases, even regulation of the process. On-line procedures in most cases must be precisely matched to the process, and the expense in terms of equipment and investment is disproportionately higher. The accuracy of laboratory analyses in the case of off-line procedures cannot be produced by using on-line procedures. There are as yet few on-line procedures for chemically analyzing solids. Near-infrared spectrometers fitted with fiber-optic sensors are used solely in the field of foodstuffs and for on-line processes. There are as yet few on-line procedures for chemically analyzing solids. Near-infrared spectrometers fitted with fiber-optic sensors are used solely in the field of foodstuffs and for on-line processes. Their sealed construction also enables material to be conditioned, e.g., humidified, granulated, dried, or rendered inert, as well as mixed. In gravity mixers, granular material is simultaneously drawn off by a system of tubes at various heights and radial locations, brought together, and mixed. Other types of construction use a considerable takeoff tube into which the solids travel through openings arranged at various heights up this pipe. If the quality of the mix does not meet requirements, the withdrawn material is fed back into the bunker (Eichler and Dau, Geometry and Mixing of Gravitated Mixtures, 1997, Proceedings, 2, 971–974). In this fashion the bunker's entire contents are recirculated several times and then homogenized. The material drawn off in most cases is delivered to the top of the bunker by air pressure (using an external circulation system).

Gravity mixers are designed for free-flowing powders and are offered in sizes ranging between 5 and 200 m³. The specific energy consumption, i.e., the energy input per product mass, is very low at under 1 to 3 kWh/t. Silo screw mixers are silos with a special funnel mixer at their outlet and are grouped with the gravity mixers. A concentric double cone gives a different residence time period for the material in the inner and outer cones, inducing remixing. Such mixers are available for quantities of material between 3 and 100 m³. In the case of granulate mixers, material from various areas of the vessel is brought together in its lower section and then carried upward by air pressure in a central pipe (using an internal circulating system) where the solids are separated from the gas and at the same time distributed on the surface. Design sizes reach up to 600 m³, and the specific energy input, like that of gravity mixers, is low. The rotating screw of a conical screw mixer transports the material upward from the bottom. This screw is at the same time driven along the wall of the vessel by a swiveling arm. This type of mixer also processes both pastes and cohesive powders. The solids at the container wall are continuously replaced by the action of the screw so that the mix can be indirectly heated or cooled through the container's outer wall. It is also used for granulation and drying. Mixers of this design are offered in capacities of between 25 L and 60 m³. In blast air or air jet mixers, air is blown through jets arranged around the circumference of a mixing head placed in the bottom of the vessel. The specific air consumption is 10 to 30 N·m³/t, and the largest mixers have a capacity of 100 m³. If a fluid flowing through a bed of particles against the force of gravity reaches a critical speed (minimum fluidization velocity), the particles become suspended or fluidized by the fluid (see Sec. 17, "Gas-Solid Operations and Equipment").

Through increased particle mobility, fluidized beds possess excellent mixing properties for solids in both a vertical and radial axis. In circulating fluidized beds often used in reaction processes, this is combined with elevated heat transfer and material circulation as a result of the high relative velocities of the gas and solids. Lower fluidizing speeds to limit air consumption are generally used if the fluidized bed serves only the purpose of mixing. Furthermore, differing volumes of air are fed to the air-permeable segments installed in the container's floor which serve to distribute air. The largest fluidized bed mixers as used in cement making reach a capacity of 10³ m³. The material must be fluidizable, i.e., free-flowing (with a particle size greater than 50 μm), and dry. The specific power input lies between
1 and 2 kWh/t, but air consumption rises sharply in the case of particle sizes above 500 μm. Fluidized-bed granulators utilize the mixing properties of fluidization for granulation, atomized fluid distribution, and drying (see “Size Enlargement Equipment: Fluidized-Bed Granulators”).

Rotating Mixers or Mixers with Rotating Component

Figure 21-85 shows four categories of mixers where the mix is agitated by rotating the whole unit or where movement in the mix is produced by rotating components built into the apparatus. These mixers are classified according to their Froude number (Fr):

\[
Fr = \frac{r \omega^2}{g} = \frac{r n^2 \pi^2}{g} \quad (21-69)
\]

Here \( r \) denotes the mixer’s radius or that of the mixer’s agitators, \( g \) the gravitational acceleration, and \( \omega \) the angular velocity. The Froude number therefore represents a dimensionless rotating frequency. The Froude number is the relationship between centrifugal and gravitational acceleration. No material properties are accounted for in the Froude number: Subject to this limitation, a distinction is drawn in Fig. 21-85 between \( Fr < 1 \), \( Fr > 1 \), and \( Fr >> 1 \). Free-fall mixers are only suitable for free-flowing solids. Familiar examples of free-fall units are drum mixers and V-blenders. However, as the solids are generally free-flowing, denaturing and segregation may also occur, leading to complete separation of the ingredients. Since drums are also used in related processes such as rotary tubular kilns or granulating drums for solids, these processes may also be prone to size segregation. In some cases, this may even be intentional, such as with rotating disc granulators common in iron ore processing. Despite these risks of segregation, mixers without built-in agitators are particularly widely used in the pharmaceuticals and foodstuffs industries since they can be cleaned very thoroughly. Asymmetrically moved mixers in which, e.g., a cylinder is tilted obliquely to the main axis, turning over the mix, also belong in the free-fall category, e.g., being fertilizer drum granulation processes. Mixing is done gently. Because of the material’s distance from the central axis, high torques have to be applied by the drive motor, and these moments have to be supported by the mixer’s bearings and bed. Units with a capacity of 5000 L are offered. There are also mixers with operating range \( Fr < 1 \) where the work of moving the mix is undertaken by rotating agitators. The particles of solids are displaced relative to one another by agitators inside the mixer. This design is suitable for both cohesive, moist products and those which are free-flowing. Examples of displacement mixers are ribbon blenders or paddle mixers. Because of their low rpm the load on the machine is slight, but the mixing process is relatively slow. The specific energy input is low and lies under 5 kWW/m³.

Ploughshear and centrifugal mixers operate in a range with \( Fr > 1 \). The consequence is that, at least in the vicinity of the outer edge of the agitator, the centrifugal forces exceed that of gravity and the particles are spun off. Thus instead of a pushing motion there is a flying one. This accelerates the mixing process both radially and axially. If the ingredients still need to be disagglomerated, high-speed cutters are brought into the mixing space to disagglomerate the mix by impact. At very high Froude number ranges (\( Fr > 7 \)) there is a sharp increase in the shear forces acting on the mix. The impact load is large and sufficient to heat the product as a result of
dissipated energy. The heat is caused by friction between the mixer's 
tools and the solids as well as by friction among the solids' particles. 
As well as simple mixing, here the mixer's task is often disagglomera-
tion, agglomeration, moistening, and sintering. Such mixers are 
especially used for producing plastics and in the pharmaceutical 
industry for granulation.

Mixing by Feeding  Direct mixing of feed streams represents 
a continuous mixing process (Fig. 21-86). The solids are blended by 
metering in each ingredient and bringing these streams of solids 
together locally. There is no axial mixing (transverse or back mixing), 
or as such it is very low, with the result that the quality of the meter-
ing determines the mix's homogeneity. Metered feeder units should 
therefore ideally be used, preferably operated gravimetrically with 
appropriate feedback control of weight loss. According to the 
requirements of the case in question, mixing is also required 
oblquely to the direction of travel. If the ingredients are brought 
together in a perpendicular fall, this is achieved by their merging 
together. If this oblique mixing is not sufficient, static mixers 
can be used for free-flowing powders or granules where, e.g., the stream 
of solids is repeatedly divided up and brought back together by baf-
fles as it drops down a tube. The energy input into the mixer is very 
low, but such systems need sufficient height to achieve mix quality.

FIG. 21-86 Direct mixing of feeder streams.
It was shown that the efficiency for radial mixing depends on the gas phase as well (O. Eichstätt, Continuous Mixing of Fine Particles within Fluid Dynamic Vertical Tube Mixers, Dissertation, in German, ETH-Zürich, 1997). At best they operate with low volume concentration and for particles between 20 and 200 μm. Static mixers have been used for very abrasive free-flow materials such as silicon carbide. Since any rotating equipment is avoided inside static mixers, abrasion is limited. As will be shown below, mixture quality is dependent on feed consistency and residence time within the static mixer. Since the latter is very short in static mixers (seconds or fractions of a second), short-time feeding precision has to be very high to achieve high-quality mix.

**DESIGNING SOLIDS MIXING PROCESSES**

**Goal and Task Formulation** An essential prerequisite for the efficient design of a mixing process is a clear, exact, and comprehensive formulation of the task and objective. Applying Table 21-22a as a checklist guarantees a systematic formulation of the mixing task along with the major formative conditions. Priority objectives covering the economic requirements, quality targets, and operating conditions have to be met when one is engineering a mixing system. Besides a definition of the stipulated quality of the mix and an average production throughput (minimum or maximum), the **quality target** can include additional physical (moisture, grain size, temperature) and chemical properties required of the mixed product. Furthermore, the general principles of quality assurance frequently demand production documentation. This means that material batches must be coded, mixture recipes recorded, and the flow of materials in and out balanced out against their inventories and consumption. Clearly formative economic conditions such as investment, maintenance requirements, and utilization of existing space often determine the actual technical features of a design when it is put into practice. Specifications arising from the mixing system's operation are grouped under formative operating conditions. These set the requirements on

- Staff numbers and training
- Process monitoring, process management system design, and the degree of automation
- Operating, cleaning, and maintenance
- Safety, dust, explosion, and emission protection and the alarm system

Sometimes raw material costs exceed the processing cost by far; or manufacturing contributes a negligible part of the overall cost; e.g., the marketing and R&D determine the manufacturing cost of a newly patented pharmaceutical product.

**The Choice: Mixing with Batch or Continuous Mixers** Mixing processes can be designed as a batch or a continuous process.

**TABLE 21-22a Checklist for Formulating a Mixing Task**

<table>
<thead>
<tr>
<th>Mix recipes (mixture composition)</th>
<th>• Number and designation of the recipes</th>
</tr>
</thead>
<tbody>
<tr>
<td>The preparation’s composition</td>
<td>• The ingredients’ percentages and margins of accuracy to be observed, particularly in the case of low-dosage ingredients</td>
</tr>
<tr>
<td>(the ingredients’ percentages and margins of accuracy to be observed, particularly in the case of low-dosage ingredients)</td>
<td>• The percentage of each recipe as part of the total production output</td>
</tr>
<tr>
<td>• The frequency with which the recipe is changed and any desired sequence</td>
<td>• Cleaning operations when a recipe is changed (dry, wet, cleaning in place (CIP))</td>
</tr>
<tr>
<td>• Sampling and analyses</td>
<td>• Sampling and analyses</td>
</tr>
<tr>
<td>Ingredients</td>
<td>• Sampling and analyses</td>
</tr>
<tr>
<td>• Designation</td>
<td>• Sampling and analyses</td>
</tr>
<tr>
<td>• Origin, supplier, packaging</td>
<td>• Sampling and analyses</td>
</tr>
<tr>
<td>• Bulk density, solids density</td>
<td>• Sampling and analyses</td>
</tr>
<tr>
<td>• Grain size (mean grain size)</td>
<td>• Sampling and analyses</td>
</tr>
<tr>
<td>• Temperature, sensitivity to thermal stress</td>
<td>• Sampling and analyses</td>
</tr>
<tr>
<td>• Sensitivity to mechanical stress (crushing, abrasion, fracture)</td>
<td>• Sampling and analyses</td>
</tr>
</tbody>
</table>

**TABLE 21-22b Comparison of Discontinuous and Continuous Mixing Processes**

<table>
<thead>
<tr>
<th>Implementation data</th>
<th>Discontinuous</th>
<th>Continuous</th>
</tr>
</thead>
<tbody>
<tr>
<td>The number of ingredients</td>
<td>As many as wanted</td>
<td>2–10; any more ingredients are usually combined in a premix</td>
</tr>
<tr>
<td>Frequency with which the recipe is changed</td>
<td>Several times per hour</td>
<td>A recipe must remain unchanged for several hours</td>
</tr>
<tr>
<td>Cleaning frequency or idle time</td>
<td>Several times a day</td>
<td>Once a day or less</td>
</tr>
<tr>
<td>Production output, throughput</td>
<td>Any rate</td>
<td>More than 100 kg/h. Exception: feeding laboratory extrusions</td>
</tr>
<tr>
<td>Risk of separation</td>
<td>Present, therefore there must be short transportation paths, few intermediate silos</td>
<td>Low risk when the material is taken directly to the next processing stage or directly drawn off</td>
</tr>
<tr>
<td>Spatial requirement</td>
<td>Large amount of space and intermediate silos required for machines with a throughput greater than 5000 kg/h</td>
<td>Low spatial requirement even for machines with a high throughput</td>
</tr>
<tr>
<td>Requirements placed on the equipment</td>
<td>Simple feeding but high demands on the mixer</td>
<td>Accurate continuous feeding (feeding scales necessary) but low demands on the mixer</td>
</tr>
<tr>
<td>Safety</td>
<td>Steps have to be taken in the case of materials with a risk of explosion</td>
<td>The small quantities of material present during processing have a low potential risk, which simplifies safety design</td>
</tr>
<tr>
<td>Automation</td>
<td>Variable degree of automation</td>
<td>Contained in the processing</td>
</tr>
</tbody>
</table>
Table 21-22b gives a detailed comparison of discontinuous and continuous mixing processes, to help guide the selection of a mixing method.

**Batch Mixing** Batch or discontinuous mixing is characterized by the fact that the mixer is filled with the ingredients, and after a certain mixing time the mixture is discharged. The feeding (or filling), mixing, and discharging operations are performed one after the other. Batch processing presents advantages for small quantities of material because of its lower investment costs and greater flexibility. Batch mixers are used even when very large volumes of material are being homogenized since continuous mixers are limited by their lower volume. However, in the batch mixer's very flexibility lies the danger that it is not being optimally utilized. For example, overmixing can occur, whereby the product could be damaged and the process's effectiveness suffers.

**FIG. 21-87** Classical automated batch mixing installation. The components are stored in small silos shown at the top of picture. The materials are extracted from these hoppers in a downstream weighing hopper according to the recipe. Once all components are fed into this weighing hopper, a valve is opened and the exact batch falls into the downstream batch mixer.

**FIG. 21-88** Weighing hopper with additive weighing for feeding a batch mixer. 1.1 Storage silos; 1.2 big bag, bag, drum; 2.1–2.2 dischargers; 3.1–3.3 feeder units; 4 cutoff; 5 flexible connections; 6 weighing hopper; 7 support for gravity force; 8 gravity-operated sensor (load cell); 9 set point; 10 weighing analysis and regulation; 11.1 measured value indicator or output; 11.2 recorder (printer); 12 cutoff; 13 flexible connection; 14 mixer; 15 discharger; 16 dust extraction and weighing hopper ventilation; 17 mixer ventilation.

**FIG. 21-89** Continuous mixing for the production of Muesli: Continuous gravimetric solids feeder (loss-in-weight feeding) supplies the components (raisins, flakes, etc.) at constant rate onto a belt which delivers the components to the continuous mixer (bottom of the picture). The continuous mixer discharges onto a second belt.
Feeding and Weighing Equipment for a Batch Mixing Process

The number of mix cycles multiplied by the usable mixer capacity gives the set mixture output per hour. The mix cycle consists of the filling, mixing, discharge, and idle times (Fig. 21-87). To this is added in special cases the time taken for sampling and analysis and that for associated processes such as disagglomeration and granulation. The capacity (throughput rate) of a batch mixing process having a mixture charge with a mass \( M \) is shown in Eq. (21-70):

\[
\dot{m} = \frac{M}{t_f + t_m + t_d + t_i} \quad \text{kg} \text{ s}^{-1}
\]  

(21-70)

The mixing time \( t_m \) depends on the selected mixer design and size, the filling time \( t_f \) on the system’s configuration, while the discharge time \( t_d \) depends on both the mixer’s design and the system’s layout. The choice of feed and weighing devices is determined by the number of ingredients, their mass and proportions, the throughput volume, the stocking and mode of delivery, the spatial circumstances, degree of automation, etc. In the simplest case the ingredients are manually weighed into the mixer. In some cases, sandwiching of specific ingredients may be desirable, i.e., staged delivery of multiple layers of key ingredients between other excipients. Where there are higher

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**FIG. 21-90** The continuous mixing of two ingredients: Axial mixing or dispersion shows up as well as residence time distribution of the product inside the mixer.

**FIG. 21-91** Dampening of feed fluctuation in a continuous mixer—variance reduction ratio (VRR). The efficiency of continuous mixing processes is described by the variance reduction ratio. The variances in concentration of inlet and outlet are compared. Tracer-feed oscillating with different periods \( T_p \), main component feed at constant rate \( 20 \, \text{g/s} \), mean residence time in the continuous mixer \( t_v = 44 \, \text{s} \). (a) Variation in time of SiC concentration: dotted line at the entrance of the continuous mixer, bold line at the outlet of the continuous mixer. (b) Power density spectrum of SiC concentration. High variance reduction ratios are achieved if the period of the tracer feed is small compared to the mean residence time in the mixer.
requirements in respect of accuracy, safety, and recording, a hopper scale represents a simple device for weighing and releasing the components into the mixing equipment (Fig. 21-88).

Continuous Mixing In a continuous mixing process (compare Figs. 21-89 and 21-90) the ingredients are continuously fed into the mixer, then mixed and prepared for the next processing stage. The operations of feeding, mixing, and discharging follow each other locally but occur simultaneously. In continuous mixing, the weighing and filling of a batch mixer are replaced by the ingredients’ controlled continuous addition. The blending time in a continuous mixer is in fact the material’s residence time, which is determined by the feed rate to the mixer. Losses of product during start-up or shutdown added to this lower degree of flexibility come as further disadvantages of the continuous process. Yet it possesses considerable advantages over batch processing both in financial terms and in respect of process control: Even high-throughput continuous mixers are compact. A smaller-volume scale provides short mixing paths and ease of mixing. When integrated into a continuous production system, a continuous mixing process saves on reservoirs or silos and automating the course of the process is simplified. In the case of dangerous products or base materials, there is less potential risk with a continuous process since only a small quantity of material accumulates in the mixer. Segregation can be limited in a continuous mixer by its smaller required scale. A continuous mixer, which on account of its compact construction can be positioned before the next station in the processing chain, guarantees that a mix of a higher quality will in fact be made available to that next stage of the process, with smaller material handling distances.

The continuous mixer has principally two tasks (Fig. 21-90): The ingredients, which in an extreme case arrive in the mixer side by side, have to be radially mixed (r). In this case radial means lateral to the direction of the material’s conveyance into the mixer. If in addition there are large feed rate fluctuations or the ingredients are themselves unhomogenized, the mixer must also minimize any differences in concentration in an axial direction (z), i.e., in the direction of the material’s conveyance, or the mixture must be axially mixed as well. If a mixer only has to perform its task radially, it can have a very compact structure, since slim-line mixers with a high rpm very quickly equalize concentrations radially over short mixing paths. Feed fluctuations (see Fig. 21-91) are damped by the residence time distribution of the material inside the mixer [R. Weinbäüer and L. Reh, Continuous Mixing of Fine Particles, Part. Part. Syst. Charact., 12, 46–53 (1995)]. The residence time distribution describes the degree of axial dispersion occurring in the mixer. The Peclet (Bodenstein) number Bo (Fig. 21-80) characterizes the ratio of axial transport velocity and axial dispersion coefficient D. The capability to reduce incoming fluctuations (thus variance) inside continuous mixers depends on the ratio of period of entrance fluctuation to the mean residence time as well as the residence time distribution. Besides the number of ingredients in the mix, a decisive feature in selecting the process is the individual component’s flow volumes. Since the feed’s constancy can only be maintained with a limited degree of accuracy at continuous feeding rates below 300 g/h, ingredients with low flow volumes necessitate a premixing operation. There is an increasing trend toward continuous mixing installations. Widely used are continuous processes in the plastics industry, detergents, and foodstuffs. Although less common, pharmaceutical processes utilizing continuous mixing are growing in appeal due to the small volume of the apparatus (see Fig. 21-92). The U.S. Food and Drug Administration, e.g., has promoted a Process Analytical Technology (PAT) Initiative with the objective of facilitating continuous processing to improve efficiency and manage variability (http://www.fda.gov/cder/ops/PAInitiative.htm; Henry Berthiaux et al., Continuous Mixing of Pharmaceutical Powder Mixtures, 5th World Congress on Particle Technology, 2006; Marcos Llusa and Fernando Muzzio, The Effect of Shear Mixing on the Blending of Cohesive Lubricants and Drugs, Pharmaceutical Technol., Dec. 2005).