Slight changes in pumping rate do not materially change dissolution characteristics. With Product A, the amount of drug in solution at the end of 3 hr. was approximately the same (85 to 90 of the 100 mg. claimed on the label) even though the pumping rate was varied from 50 to 70 ml./min.

The operating characteristics of this apparatus can be easily altered. However, when conditions are fixed, similar dissolution profiles are obtained when drug or tablets from a uniform lot are subjected to the procedure. Repeated tests with Product A[1] produced dissolution profiles similar to that shown in Fig. 3. Moreover, the basket-stirrer assembly positions the tablet in the same way from run to run and helps, therefore, to produce results which are characteristic of the product rather than the apparatus.

[1] Butazolidin tablets, Geigy, Ardsley, N. Y.

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Keyphrases

Continuous flow apparatus—tablet, capsule dissolution
Dissolution profile determination—apparatus
Diagram—continuous flow dissolution apparatus

Some Measurements of Friction in Simple Powder Beds

By EVERETT N. HIESTAND and CHARLES J. WILCOX

An apparatus and procedure for measuring the static friction coefficient of powders in simple beds are described. The relative merits of three variations of the shear cell are considered. A sandwich of powder between two surfaces covered with sandpaper is satisfactory under most experimental conditions. Examples of the effect of relative humidity, time under load, mechanical vibration of the powder bed, and variations in the experimental procedure are presented. Reproducible values of the friction coefficient can be obtained readily if the experimental and environmental conditions are standardized. The results of the experiments suggest that a single value of the friction coefficient applies only to a specific powder bed. In practical applications the entire range of values that may be encountered must be within acceptable limits.

Friction coefficients and cohesion values have been used to characterize the flow properties of powders. In a recent review (1), the methods used and the problems encountered have been discussed. In the opinion of the authors, there are limitations on the usefulness of such data because neither the friction coefficient nor the cohesion is a single valued property of the powder. As stated in the review article, "The properties of a powder bed depend on the cumulative effect of the previous history of all the portions of the bulk being considered. Isolated regions of shear, vibration, or compaction may have produced high bulk density regions. These may remain intact in subsequent flow of the powder or may fragment into macroscopic regions mixed throughout the less dense bed. The forces acting on the top of the bed may be quite different from those at the bottom of the bed." In the studies reported here, considerable data have been collected that reveal the large number of factors that cause changes in the properties of powder beds as well as indicating the magnitude of these effects.

The basic measurements of friction coefficient and cohesion may be obtained from a simple shear cell described by Nash *et al.* (2). Though

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modified in detail, the apparatus used in these studies retains the advantages of simplicity and the use of a thin layer of powder that may be formed by gentle sifting into place. Deep powder beds are not necessary and may be undesirable since uniform bulk density is difficult to maintain in forming deep beds. Other shear cells have been used successfully but some of the observations reported here could not be made using them because of the pretreatment, packing, *etc.*, of the powder bed (3).

FRICTION COEFFICIENT AND COHESION

The friction-cohesion equation used here is basically the same as suggested by Deryagin (4).

$$\tau = \mu\,(mg + c) \qquad \text{(Eq. 1)}$$

where τ is the force required to shear the powder, μ is the friction coefficient, mg is the load in dynes applied normal to the shear plane, and c is the apparent cohesion force of the powder.

Since friction is said to be the shear strength of the areas of true contact, an increase in friction coefficient should reflect either an increase in true contact area or an increase in force of interaction in the regions of true contact. Either of these should produce a corresponding increase in cohesion. In real cases the cohesion value obtained by applying Eq. 1 often decreases when the friction coefficient increases and vice versa. These experimental values vary in such a way that the authors have been unable to determine their significance. Other workers have observed that values of c based on Eq. 1 are larger than values obtained by direct tensile strength tests (5).

APPARATUS

Shear Cells—The basic shear cell is in the form of a sandwich of powder between upper and lower plates. The powder bed is prepared by sifting it into the 6.2-cm. diameter hole of a thin template placed over the bottom plate. The thickness of the template used varies with the type of upper and lower plates employed. A spatula serves to level the surface by gently scraping away excess powder. Care is taken to accomplish this with minimum disturbance of the powder bed. The template is lifted off and a disk-shaped bed of powder remains. The top plate is gently lowered onto the powder bed to bring its surface into contact with the powder. Additional weights are added to produce the desired applied load. Figures 1 and 2 show the cell in place in the apparatus. Normal loads less than the weight of the top plate are produced by suspending the top plate from one side of the analytical balance holding the desired counterbalance weights (not shown in Figs. 1 and 2). In this case the powder bed is gently raised to meet the top plate and to zero the balance.

Several different designs of top and bottom plates have been used. For some experiments at smaller applied loads, the bottom plate was a rectangular piece of "harrow" assembled from file cards[1] after reducing the bristle length. The top plate was a 5-cm. diameter "harrow" of the same material. This shear cell "sandwich" will be referred to as the HPH cell (harrow-powder-harrow). At higher loads, the harrow teeth may touch during a determination. Consequently, the upper and lower plate materials were changed. The first experiments done in this laboratory used metal disks coated with sandpaper,[2] called the SPS cell. In most of the recent studies the plates have been made from compressed tablets[3] prepared from the same powder used in the shear test, the TPT cell. Figure 3 shows the parts of these three types of cells. The top plate has been removed and placed upside down beside the powder bed.

In a few cases, a direct tablet-to-tablet measurement was made, the TT cell. This would not be a

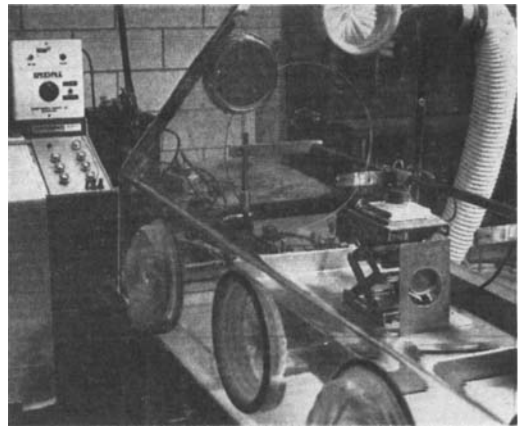

Fig. 1—Apparatus, TPT shear cell in place.

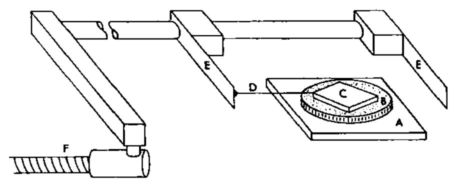

Fig. 2—Schematic arrangement of apparatus, Key: A, *bottom plate of cell;* B, *powder bed;* C, *top plate of cell;* D, *tow line;* E, *strain gauges (only one used at a time);* F, *jack.*

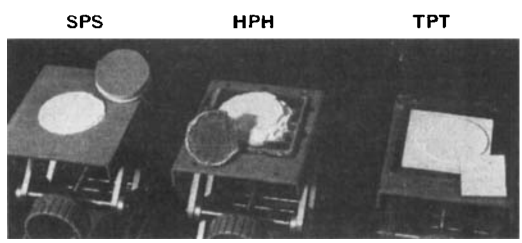

Fig. 3—Three varieties of shear cells used.

[1] Coulton's No. 10 File Cleaner, E. C. Knudson, Mfg., Chicago, Ill. Facing removed and cemented to flat plate. Surface grinder used to reduce length of steel bristles to 0.16 cm.

[2] The grade of sandpaper is not a critical factor. A satisfactory paper is garnet finishing paper, open coat, No. 100, Minnesota Mining and Mfg. Co., St. Paul, Minn.

[3] The top element is a tablet 3.7-cm. square and about 0.64-cm. thick. The bottom element is four of these tablets cemented to a rigid base plate and sanded smooth. Each tablet was made from 10 g. of powder A special die was used with the Carver press to form the tablets. Care was taken to make all tablets in a set as nearly alike as possible.

powder study unless indigenous powder was present. However, a comparison of TPT and TT data was desired.

Force Detector—A cantilever beam strain gauge was connected to a horizontal screw jack. The jack was driven through a gear box by an electronically speed-controlled, reversible electric motor. The upper plate of the shear cell was connected to the strain gauge by a small diameter tow line or wire including a fish-line swivel. The strain gauge output was connected directly to the input of a Sargent model SR recorder and resulted in a sensitivity of 1.315×10^6 dynes/mv. These parts are shown in Fig. 1 and diagrammatically in Fig. 2.

The usual linear speed of the gauge assembly was 4 cm./sec. Only static friction or "breakaway" force values were used. The shear force was insensitive to the rate of application of force in this range of gauge velocities.

The usual operating procedure was to assemble the cell and connect the linkage to the gauge. The screw jack was operated until the top plate moved. Then the direction of the jack was reversed until the tow line became slack. The forward motion of the jack was reestablished and a second pull was made. This sequence was repeated until the same shear force was observed for several sequential pulls. This was called the plateau value of the shear force and the powder bed was considered to have attained a reproducible steady-state condition referred to as the plateau condition.

The frequency of the pulls was arbitrarily selected as approximately 2/min. The loading and unloading times vary with the magnitude of the shear force and therefore with the applied load. The time interval between the "breakaway" peak force and the unloading is determined by the backlash of the jack. The exact magnitude of these factors is not critical but uniformity of practice is desirable to reduce the variability of results.

Environment—The shear cell assembly was contained in a controlled-humidity chamber. The humidity chamber draws from dry and wet air reservoirs to cycle the relative humidity over a range of about $\pm 1\%$ every 2 min. The air in the chamber is circulated rapidly through the chamber and a mixing box beneath it which contains the humidity controller and the dry and wet air-injector systems.

EXPERIMENTAL MATERIALS

With a few exceptions, the materials used were selected from the common chemical entities used in pharmaceutical dosage forms. They were preconditioned in the controlled humidity environment for no less than 24 hr. and usually for more than 80 hr. before use in the shear cell. The dynamic nature of the properties of powders will be illustrated by observations of the changing of a sample over very long periods of time.

CHANGES OF SHEAR STRENGTH IN A SERIES OF PULLS

The series of pulls used to establish the plateau condition will provide a pattern of shear values typical of the powder bed at the applied load. In general, one of three patterns is obtained. These

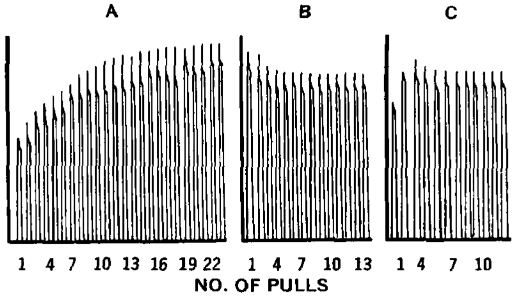

Fig. 4—Recorder chart traces. Patterns developed by peaks prior to reaching plateau condition.

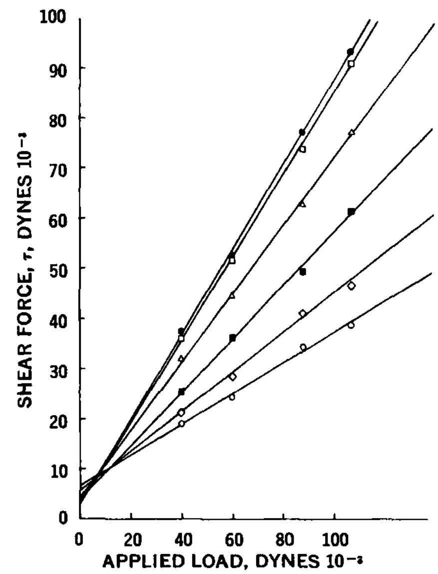

Fig. 5—Repeated shearing of powder bed changing its properties. Each line represents the shear force for a given number of pulls on the top disk obtained at various applied loads. The HPH cell was used in 50% relative humidity environment with calcium acid novobiocin powder. Key: ○, *1st pull;* ◇, *2nd pull;* ■, *4th pull;* △, *8th pull;* □, *16th pull;* ●, *plateau.*

are shown in the recorder chart traces in Fig. 4. The peak value of the leading edge of each individual "spike" is the breakaway force. Once shear occurs, the measurement becomes one of dynamic friction and not static friction. The "spike" ends when the force is removed by reversal of the jack. The regular spacing of these spikes shows the uniform schedule of the pull cycles. This is necessary to obtain reproducible results.

One of the patterns shown in Fig. 4 is obtained at each individual applied load. The usual friction plot used in this work is obtained by plotting the plateau values of the shear force against the applied load. Others have preferred to use the first pull data (2). Figure 5 shows both results with calcium acid novobiocin as well as plots obtained by using other selected values, *e.g.*, only second pull values for each series, only fourth pull values, *etc.* At all loads used, the pattern of pulls was like those in Fig. 4A. All strongly cohesive powders produce patterns of the type in Fig. 4A. These changes with shear may be produced by changes in packing density, changes in bed thickness, or a development of structure that resists particle movement in a given direction. It is not the result of powder piling-up in front of the top element of the cell since removal of the powder in front has negligible effect on the shear force for the following pull. The types of patterns in Figs. 4B and 4C probably result from powders in which particle orientation and/or dialation are dominant factors associated with shearing of the bed. The bed thickness of choice depends on the selection of the shear cell. Obviously the HPH cell must use thicker beds than might be used with the TPT arrangement. Separate studies using beds of different initial thickness gave very similar results, though not always identical values of τ and c. With the HPH cell, the plateau values remained constant until the teeth of the harrow made contact with each other. With the other cells the plateau values remained constant over many pulls. Therefore, changes in bed thickness are not believed to be major factors in these measurements, especially when plateau conditions are used. Probably, the plateau condition is a steady-state situation in which the rate of introduction of new powder at the leading edge remains nearly constant with each successive movement of the top plate (see section on the elasticity of the tow line).

If one concludes that the changes observed in a series of pulls is the result of changes in the bed other than thickness *per se*, then one may conclude that in some applications the friction and cohesion values may vary over at least the range observed in the series of pulls. Certainly a single value of friction and cohesion cannot be characteristic of the powder but only of the specific powder bed. It would be interesting to relate these values to other properties such as bulk density, *etc.* However, it is not simple to obtain both measurements on the powder in the cell under the conditions of each individual pull.

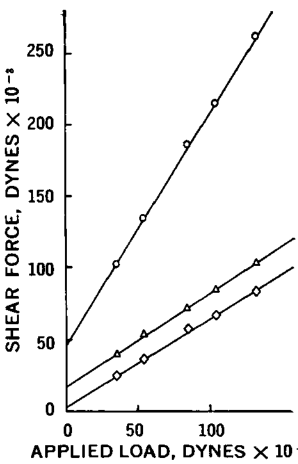

Fig. 6—Lidocaine base, SPS data, 70% relative humidity; influence of time of static loading on magnitude of shear force for first pull. Key: ○, *1st pull after 30 min.;* ◇, *2nd pull, immediately after 30-min. delay pull;* △, *plateau by usual method.*

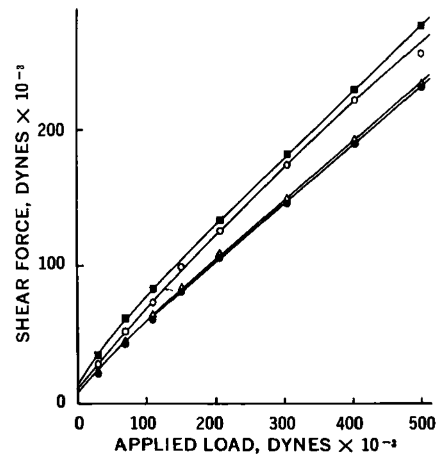

Fig. 7—Chlorphenesin carbamate, TPT data, 50% relative humidity; influence of time of static loading on magnitude of shear forces after plateau condition has been established. Key: ■, *1st pull after 60-min. wait;* ○, *1st pull after 30-min. wait;* △, *plateau;* ●, *2nd pull immediately following 1st pull after 30-min. wait.*

TIME-DEPENDENT EFFECTS

Duration of Application of Static Load—It has been reported that the static friction coefficient between solids varies with the logarithm of the time of contact (6). There is no reason to expect the friction coefficient of powder beds to be independent of time of contact. Lidocaine base illustrates an extreme case as shown in Fig. 6. When the load was placed on the top plate and left for 30 min. before the first pull was made, the static friction force was much larger than when the usual time schedule was followed. However, the next pull, made immediately after the time-delayed pull, is only very slightly different from the first pull made on the usual schedule without the time delay.

Similar results were obtained when the time studies were applied to powder beds brought to the plateau condition. Figure 7 shows data obtained with chlorphenesin carbamate. The plateau condition was established and then the cell was left undisturbed for the chosen time interval. The magnitude of the first pull after the time interval was higher than the plateau value. The lowest line in Fig. 7 shows the magnitude of a second pull made immediately after the time-delayed one for the 30-min. case. Similar results were observed after each of the time-delay intervals. The shear forces have returned to approximately the same as the plateau condition.[4] This indicates that the changes in shear force with time of static load application are not due to irreversible changes in the power bed.

Figure 8 shows a longer interval of time lapse than shown in Fig. 7 but for only one applied load. Note that changes still are occurring after 30 hr. of standing with the load applied.

Again the conclusion is that a single value of the friction coefficient and the cohesion is not adequate to describe the powder properties.

Effect of Storage Time in the Controlled-Humidity Chamber—Changes in the properties of powders

[4] The fact that this line is slightly below the plateau value in both examples is consistent with an observation made in other cases, *viz.*, whenever a preceding pull results in an unusually high shear force the next pull will be smaller than the average value.

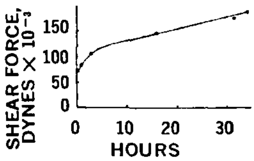

Fig. 8—Chlorphenesin carbamate in the same study as in Fig. 7 but shows extended time scale for the application of a 110-g. applied load.

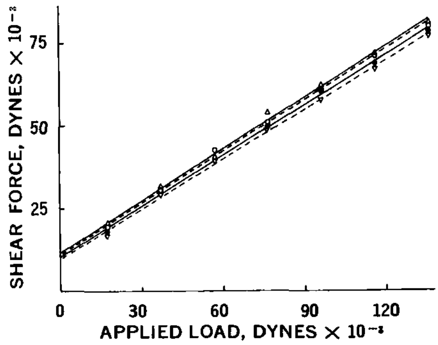

Fig. 9—Changes in the properties of a placebo mixture stored in the controlled-humidity chamber. Key: △, 0 days; ○, 3 days; ●, 14 days; ▽, 25 days.

occur during the storage of the material in the controlled-humidity environment. Figure 9 shows the results of a series of measurements[5] with the HPH cell on a powder mixture. The mixture was used for a placebo tablet preparation and its primary constituent was lactose. The powder was not preconditioned in the controlled-humidity environment. Zero time is taken as the time when the powder was spread on a tray in a layer not in excess of 1.2 cm. thick and was placed in the controlled-humidity chamber for the duration of the experiment. The results of the study indicate that it did not attain an equilibrium with its environment in 25 days.

The controlled environment limits the humidity fluctuations to a range of ±1% relative humidity. Moisture on the surface and especially fluctuations in the amount of moisture could promote surface changes of the solid. However, the environmental changes in relative humidity are rapid enough that one would expect very little effect. Certainly one would not expect all powders to continue to change over such long periods of time but this example illustrates that the possibility must be considered.

EFFECT OF RELATIVE HUMIDITY OF THE ENVIRONMENT

As indicated earlier, most measurements were made after powders had been stored for reasonable lengths of time in the controlled-humidity environment. The option of ignoring the effects of time of storage of each material was adopted, obviously to save time. This expediency detracts from the absolute precision of a given value but should not mask the important effects.

Table I shows the differences in plateau values of the friction coefficient obtained in various relative-humidity atmospheres using the TPT cell prepared from β-sitosterol. A minimum value in the 25–65% relative humidity range was expected. The dramatic changes in the shear forces observed in the 30 and 50% relative humidity range were not expected.

TABLE I—CHANGES IN THE FRICTION COEFFICIENT OF β-SITOSTEROL POWDER, TPT CELL DATA

Relative humidity, %	30	40	50	72	87	94
Friction coefficient	0.91	0.76	0.87	0.93	0.94	0.89

EFFECT OF VIBRATION ON SHEAR FORCE

One possible cause of changes of shear force with time is the change in area of true contact induced by the ever present ambient vibrations of the laboratory. If so, an increase of shear force with time should be accelerated by subjecting the shear cell to forced vibration.

An electric vibrator[6] was attached to the lab jack supporting the shear cell. The motion orientation was parallel to the direction of shear. A plateau value was attained in the same manner as before except that the vibrator was turned on for a few seconds between each pull Figure 10 compares the plateau condition results with spray-dried lactose (TPT) subjected to a 4-sec. vibration period with the unvibrated case. On the same graph is shown this same comparison for the TT cell. Figure 11 shows

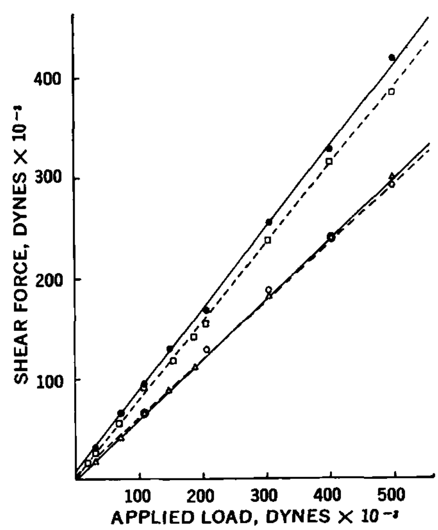

Fig. 10—Lactose, spray dried, 50% relative humidity, TPT and TT data; effects of vibration on the values at plateau conditions. Key: ●—, TPT vibrated 4 sec.; △—, TPT not vibrated; □---, TT vibrated 1 sec.; ⊕---, TT not vibrated.

[5] These data are taken from a study in which all measurements were made using a standard powder bed that reached a plateau condition with 138 g. applied load prior to shearing at the desired load.

[6] Syntron Electric Vibrator, type V-4, style 1518, Syntron Co., Homer City, Pa.

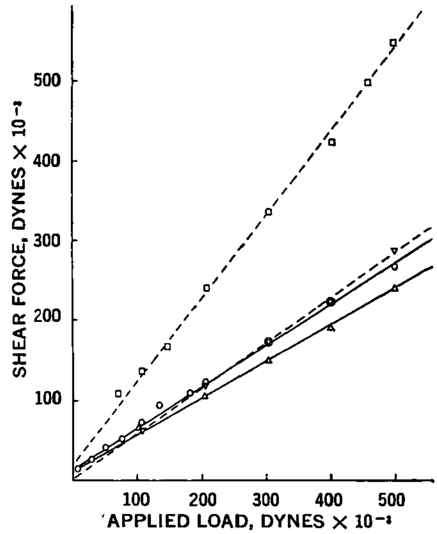

Fig. 11—Chlorphenesin carbamate, 50% relative humidity. TPT and TT data. Effect of vibrations on the values at plateau conditions. Key: □---, TT vibrated; ▽---, TT not vibrated; ○——, TPT vibrated; △——, TPT not vibrated.

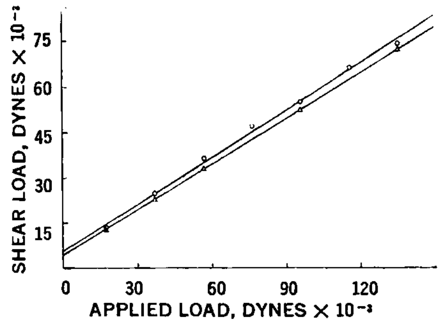

Fig. 12—Changes in the shear forces at plateau conditions resulting from the repeated sifting of the same sample of the powder in a placebo mixture. Key: ⊙, new powder; △, sifted 5 times.

Table II—Effect of Elasticity of Tow Line on Plateau Shear Values for 140-g. Applied Load

Linkage Material	Shear Value, τ_p, Dynes × 10^{-3}	Average Distance Moved per Pull, mm.
Small nylon thread	87.1	1.45
Nylon, No. 15 fish line	92.5	0.53
Wire	93.2	0.44

the analogous case using chlorphenesin carbamate.

TT data for materials such as lactose may be misleading because some powder is formed at the shear plane between the tablets. No evidence of powder was found for the TT case with chlorphenesin carbamate. Forced vibrations produced, in a few seconds, an effect similar to that produced by standing for a much longer time; however, the phenomenon occurring in the powder bed could have been different in the two cases. Possibly the main effect of vibrations is to produce packing changes. Standing for longer periods may permit only the relaxation of elastic stresses at points of true contact. Either could increase the friction force. Again it is difficult to make measurements that would define unequivocally the mechanism producing the effect.

SOME RESULTS RELATED TO EXPERIMENTAL PROCEDURES

Sifting—The same lactose placebo mixture used in the study of changes during storage was used to demonstrate the effects produced by repeated sifting. Figure 12 shows the result; the HPH method was used. Since the sifting to form a shear cell normally is done in the air bath, *i.e.*, the controlled-humidity chamber, in which there is rapid circulation of the air, some fine particles are swept away by the air currents. This is believed to be the cause of the observed changes. These results led to the use of a fresh sample of powder each time a powder bed was formed.

Elasticity of Tow Line—It is obvious that the factors that produce variation in the observed values of the friction coefficient are multitudinous. The influence of the design of the apparatus cannot be eliminated. Table II shows data obtained using three different tow lines between the cell and the strain gauge. The nylon line stretches as tension is applied. When the static friction is overcome, the top plate of the cell will move forward until the tension is reduced to the dynamic force. Obviously this distance of travel depends on the elastic properties of the tow line and the cantilever beam strain gauge assembly. In this study only the tow line was changed. Apparently, the structure of the bed of powder is dependent on the extent of movement of the top plate. Therefore, the plateau value changes. Possibly the differences result from the change in the rate of introduction of new powder at the leading edge.

Differences in Values with Various Shear Cells—It must be noted that the three cells, HPH, TPT, and SPS do not yield identical results in the shear cell studies. The HPH design was developed to assure that the shear was within the powder bed. In concept it is essentially the same as a cell used by Dawes (7) but of a much simpler design. Dawes compared the results obtained using a cell with sandpaper-coated surfaces with those using a cell with vanes extending into the powder bed. The absolute values were found to be significantly different.

The results of these studies confirm this observation. Table III gives examples. Each cell fits a specific need and also has specific limitations. The HPH cell produces shear in the powder bed but cannot be used to obtain plateau conditions if a large applied load is used because the teeth of the harrow-like plates will touch before the plateau

Table III—Comparison of Values Obtained with Lactose Using Different Shear Cells

	HPH	TPT	SPS
Friction coefficient, μ	0.608	0.608	0.602
Cohesion (dynes/cm.^{2})[a]	+59	−57	+260

[a] These units are obtained by dividing the intercept on the σ axis by (μ × area of top plate).

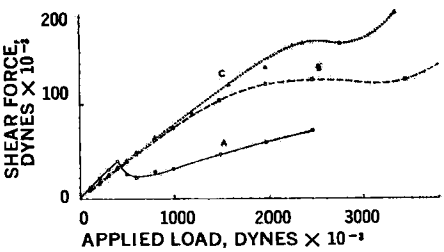

Fig. 13—Anomalous results obtained for plateau conditions with magnesium stearate. TPT and lower SPS data were obtained by pulling top plate in one direction. Top SPS data were obtained by reversing direction of pull each time. Key: A, *TPT data for* τ_p, *all pulls in same direction;* B, *SPS data for* τ_p, *all pulls in same direction;* C, *SPS data for* τ_p, *pull direction reversed after each pull.*

condition is established. The TPT cell is useful with the larger applied loads and does not introduce a foreign material at the interface with the powder. Obviously it cannot be used when the powder will not form a sufficiently strong compact by compression. Sometimes shear may occur at the tablet-powder interface more readily than within the powder bed. With magnesium stearate the surface of the tablet changed during a single run. Reproducible results were obtained by removing the surface layer between each run. Figure 13 shows three friction plots obtained with magnesium stearate. The conditioning of the tablet surface and shifting of the shear plane to the interface is believed to account for the irregular shape of the plot. The SPS cell required larger loads to produce the effect. After shearing to the plateau condition under these larger loads, the sandpaper was plated with magnesium stearate and appeared to be just like the compressed tablet surface. Pulling the top plate alternately in opposite directions diminished this effect and delayed the onset of gross deviations in the plot. The SPS cell seems to be the most versatile assembly.

Differences Produced by Direction of Shear— If after a series of pulls in a given direction the direction of pull is reversed, the observed shear force changes to be more nearly like the first pull, *e.g.*, powders producing patterns like Fig. 4A would require a smaller shear force when the direction of shear is reversed, if like Fig. 4B a larger force would be required. Table IV lists plateau values for several powders. The usual one direction of pull values are given and also the values obtained when the direction of pull is reversed after each individual pull. The magnitude of the plateau values seems to follow the same general rule as for the case of a single pull in the opposite direction.

The failure of these experiments to produce values independent of the detailed design of the apparatus and independent of the procedure followed in its use could prompt extensive criticism

Table IV—Comparison of Plateau Values of Shear Force Between Case of All Pulls in Same Direction, τ_p, and Case of Pull Direction Reversed After Each Pull, $\tau_p \rightleftarrows a$ (SPS Cell, 50% Relative Humidity)

Material	Plateau Value of Shear Force τ_p	$\tau_p \rightleftarrows a$
β-Sitosterol[a]	0.90	0.79
Lactose (spray dried)	0.63	0.65
Erythromycin base	0.82	0.72

[a] Different lots of drugs were used than for studies reported in Tables I and II.

of the entire study. However, many of the conclusions have been made using a carefully standardized procedure in which given values may be reproducibly obtained. Therefore, changes in these values produced by humidity, time, vibrations, *etc.*, are believed to be meaningful observations. The discussion of the observations relating to the experimental design or procedure may assist other investigators interested in conducting similar experiments.

CONCLUSIONS

A relative simple apparatus and procedure are described for obtaining reproducible powder bed conditions. Experimental results have been obtained that indicate some of the factors in the environmental history of a powder bed that produce changes in the friction and cohesion within the bed. In practical applications, the entire range of values of friction coefficients and cohesion should be within acceptable limits. The experimental design used to establish the significant range of values must include the use of powders subjected to the environmental variations that will be encountered by the powder in actual use.

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Keyphrases

Friction measurements—powder beds
Shear strength—powders
Static friction—powder beds
Vibration, effect—shear force
Humidity, effect—friction in powders
Diagram—friction measurement apparatus