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ACKNOWLEDGMENTS AND ADDRESSES

Received October 26, 1970, from the *Analytical Research and Development Division, Ciba Pharmaceutical Company, Summit, NJ 07901*

Accepted for publication January 14, 1971.

Impact Test for Hardness of Compressed Powder Compacts

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Abstract ☐ An impact–rebound method of estimating the hardness (*i.e.*, the pressure necessary to produce permanent deformation) of a compact of powdered solid is evaluated. A steel sphere arranged as a pendulum acts as the indenter. Since energy consumed during impact is used in doing pressure–volume work, this energy divided by the volume of the indentation provides an estimate of the mean deformation pressure. Two methods of estimating the dent volumes are compared. The simpler method uses equations adopted from metallurgy. These estimate the volume from the energy consumed during impact and the chordal radius of the dent. The alternate method estimates the volume from the displacement of a grid of lines projected onto the dented surface at a small angle of incidence. The latter method gives slightly smaller volumes than the former. When plotted in various ways, the data obtained yield slopes and/or intercepts consistent with the adopted metallurgical equations. Therefore, these equations are considered to provide: (*a*) an acceptable description of the indentation process and (*b*) the more satisfactory method of estimating the hardness of compacts. Plots of log pressure *versus* relative density appear to be linear. Extrapolations of these plots to a relative density of unity provide estimates of the properties of a single polycrystalline mass of the powdered material.

Keyphrases ☐ Powder compacts, compressed—impact test ☐ Compressed powder compacts—impact-rebound hardness test ☐ Indentation volume measurement—compressed powder compacts ☐ Diagram—pendulum impact device

In previous communications (1–3), the senior author indicated that the mechanical properties of solids are important fundamental properties which affect the flow and bonding properties of solids. Shlanta and Milosovich (4) studied some of the time-dependent effects, and Shlanta (5) reviewed the importance of plastic deformation in tableting. Huffine (6) also considered these properties. Because the anelastic properties of crystalline solids are dependent not only on the strength of the intermolecular interactions but also on the degree of imperfection of the molecular arrangement, the plastic yield pressure is not an intrinsic property of the solid state of a chemical compound in a given polymorphic form. The procedure followed in crystallizing the compound and the subsequent working of the solid may influence the plastic yield value.

Hardness may be defined as the resistance of a solid to permanent deformation (7). Indentation methods are standardized and are common practice for assessing the hardness of metals. Previous reports in the pharmaceutical literature (8–10) of the use of indentation methods have not established whether the equations used with metals may be applied to compacts of organic materials. It is the purpose of this article to explore this aspect for the dynamic, *i.e.*, the impact–rebound, method of hardness testing. The theoretical equations adopted from the metallurgical field are described later in this communication.

APPARATUS

The pendulum arrangement for controlling the pathway of a sphere is used in this apparatus. This arrangement simplifies the control and measurement of the initial and rebound height. Figure 1 shows the apparatus diagrammatically. Figure 2 is a photograph of the apparatus (an air shield was removed to expose the apparatus).

Two dies are used in these studies: one is of square cross section and the other is circular. Both have a cross-sectional area of 2.25 in.^{2}. Eight to twenty grams of powdered solid is used in the die. The compact is pressed with a hydraulic press[1]. The die is held between the punches only by the friction with the powder, thereby permitting both punches to move. To increase the probability of a series of tablets having identical properties, the pressure is raised to a selected value and maintained for a definite time. Figure 3 shows the two dies with one punch and the compact in place.

EXPERIMENTAL PROCEDURE

After removal from the press, the compact is pushed to within a couple of millimeters of the surface of the die. The die, F, containing the compact, B, and longer punch, G, are clamped into the apparatus by means of clamps, J. The back-up block, H, is moved up to the punch and its bolts are tightened only moderately. Bolt I is then used to push against the support post, N, until enough force is developed to push the back-up block, the punch, and the compact until the compact is moved flush with the die surface. The bolts on the back-up block are then tightened. Thus, only one face of the solid is left unsupported.

The sphere, A, is raised against the pointed pole of the magnetic hold, E. Both a.c. and d.c. currents are used initially to cause the sphere to vibrate against E and seek an equilibrium orientation. Before release, the a.c. signal is removed and the sphere comes to rest held by the d.c. field only. The sphere is released by opening the d.c.

[1] Model 341-20, Loomis Engineering and Manufacturing Co., Caldwell, N. J.

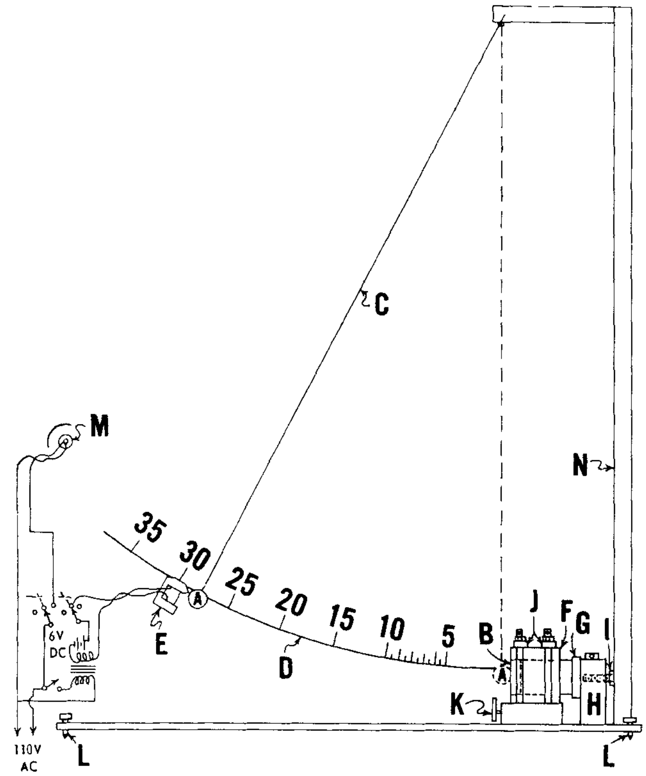

Figure 1—*Schematic of arrangement of pendulum impact device. Key: A, steel sphere; B, powder compact; C, wire support of pendulum; D, scale of angle from vertical; E, magnetic hold-release; F, die; G, punch; H, back-up block; I, back-up bolt; J, die clamp; K, stop (winch driven); L, leveling screws; M, light; and N, support post.*

circuit. At the same time a light, M, turns on and a camera shutter is opened to make a time exposure of the rebound of the sphere.

An electrically driven winch also starts when the sphere is released. With proper timing, it moves a metal stop between the compact and

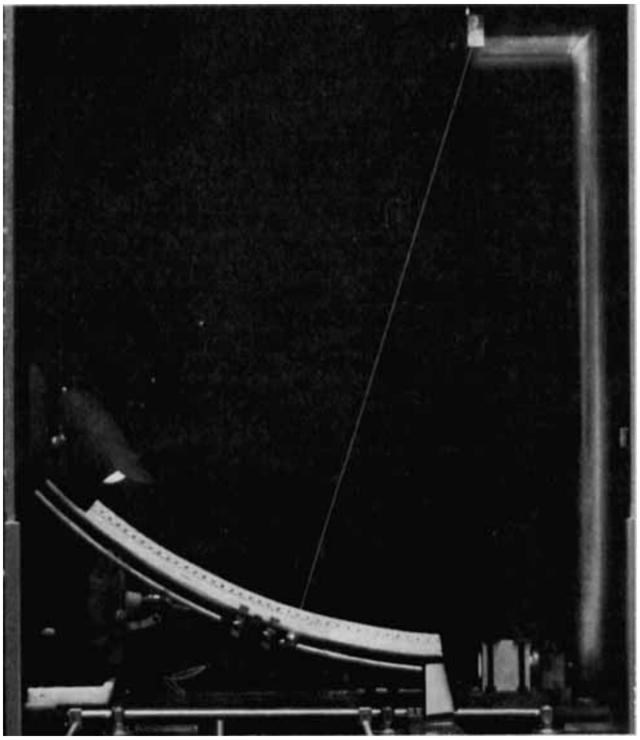

Figure 2—*Photograph of pendulum impact device.*

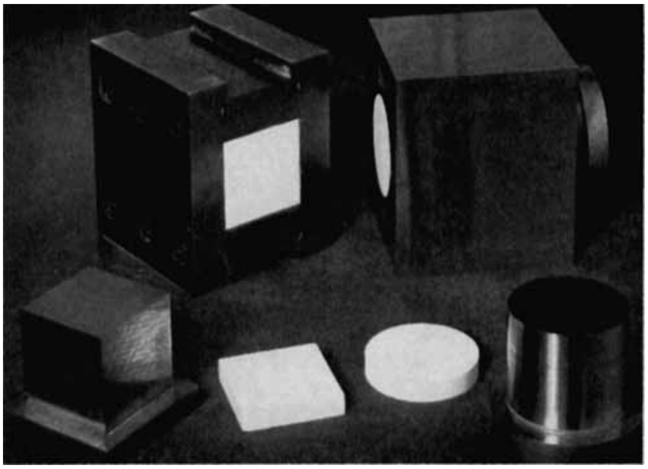

Figure 3—*Two dies and punches with compact in place as used in the impact test. A separate compact after removal from the die is shown also.*

the sphere after the rebound occurs. This prevents a second impact from occurring.

If desired, additional hits may be made on the same spot. An "off-center" hit will be evident by an "off-side" rebound. However, the magnetic hold and release arrangement nearly always puts the sphere on target.

Measurement of Indentation—The volume of the indentation may be obtained by the optical procedure outlined in a subsequent section. This procedure permits an evaluation of the volume without removing the die and compact. This feature is essential if successive hits are to be made on the same compact.

Theoretical equations are derived for calculating the volume from the initial and rebound heights of the sphere and the chordal diameter of the dent. This diameter may be obtained either by direct microscopic measurement or by measurement from an enlarged photograph. With some materials, shadowing lightly with black paint increases the contrast at the edges sufficiently to make the diameter measurement easier and more precise. Spraying at a low angle of incidence produces the shadow. A mean value of five determinations and a statistical evaluation of errors are useful in these measurements.

Estimation of Mean Deformation Pressure—The assumption of a constant mean deformation pressure[2], P, is mathematically equivalent to the calculation of the average mean deformation pressure because

$$P = \frac{\int_0^{V_p} P_z \, \delta V}{\int_0^{V_p} \delta V} = \frac{E_p}{V_p} \qquad \text{(Eq. 1)}$$

where P_z is the mean pressure for penetration z, V is the variable volume, V_p is the permanent dent volume, and E_p is the work of deformation. If all the energy lost by the sphere is assignable to E_p, then $E_p = E_i - E_e$, where E_i and E_e are the respective initial and rebound potential energies of the sphere. Therefore, $P = (E_i - E_e)/V_p$. This simple equation is all that is needed to estimate P if the volume is measured directly. Since there is no real advantage to the awkward terminology, average mean deformation pressure, only mean pressure will be used hereafter.

Equation 10 permits P to be estimated by measuring the chordal diameter, $2a$, of the dent instead of its volume. A similar equation, 12, estimates the volume of the dent. These calculations introduce additional assumptions. Before they are used, it must be demonstrated that they yield good estimates. Since both Eqs. 10 and 12 contain the same assumptions in their derivation, it was considered sufficient to test only one of them. An independent measurement

[2] The pressure is not a constant over the sphere–compact interface because of friction and stress gradients in the compact.

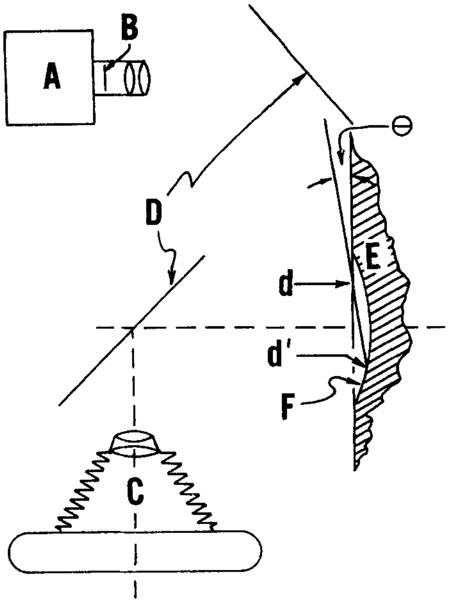

Figure 4—*Schematic of arrangement for determining dent volume. Key: A, slide projector; B, transparency containing grid of lines; C, camera; D, first surface mirrors; E, compact; F, surface of indentation; θ, angle of incidence of projected image; d, location of an image of vertical lines on undented surface; and d′, location of image of the same vertical line on dented portion of surface.*

of the volume appeared to be easier than a direct measurement of pressure.

Measurement of Dent Volume—The surface characteristics and the fragile nature of some compacts restrict the suitable methods for measuring dent volume. The following optical method was selected because it did not disturb the compact. In fact, the data are obtained without removal of the compact from the apparatus.

An image of a grid of vertical lines was projected horizontally at a small angle of incidence, θ, onto the tablet surface. The lines of the grid were spaced to produce equally spaced vertical lines on the tablet surface. At the indentation, the vertical lines became curved, the horizontal displacement being proportional to the depth of the indentation. Figure 4 shows schematically the arrangement. By photographing the tablet, the image of the grid could be recorded. Figure 5 shows a typical photograph of a grid image on a dented compact. To estimate the displacement accurately, the photograph was made with transparency film and projected onto a large sheet of plain white paper.

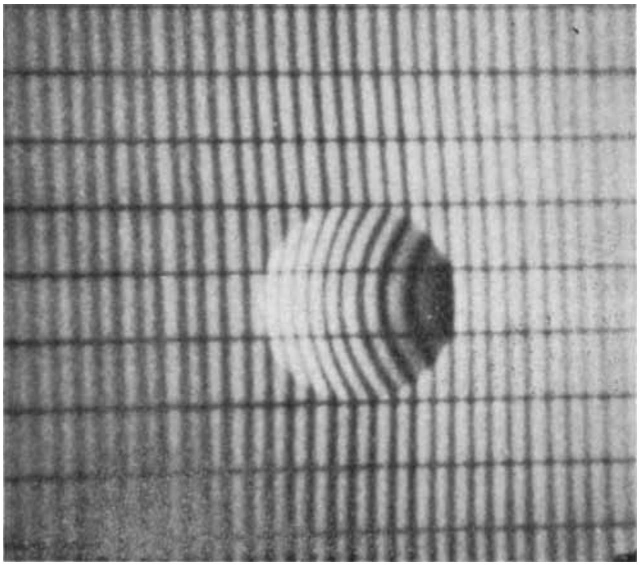

Figure 5—*Magnified photograph of grid image on compact surface. Horizontal line spacings on the compact surface are determined with a cathetometer. The photographic and projection magnification factor is determined by change in spacing of these lines.*

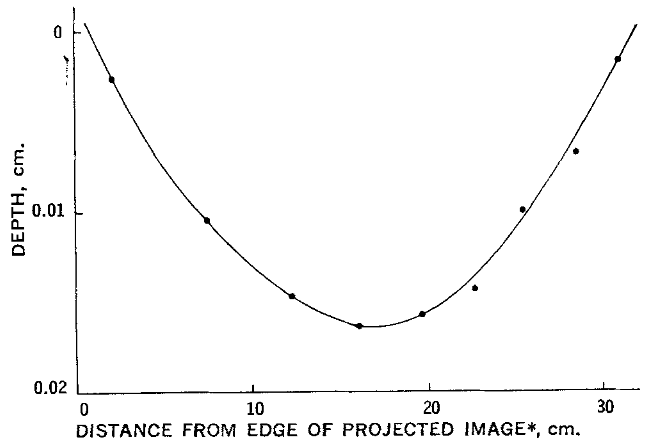

Figure 6—*Plot of data scaled from an enlarged image of the type shown in Fig. 5. * This scale is magnified 66.6 times the true dent size.*

The depths determined from the photograph were plotted against the distance from an arbitrary point near one edge of the dent. A smooth curve was drawn through the points and the center, and maximum depth was obtained from these "smoothed data." To simplify the volume calculation, the data were replotted with transposed axes. The log of the distance above the maximum depth, d, was plotted against the log of the chordal radius, α_d. These data approximated a straight line on the log–log plot. Therefore, the cross-sectional surface of the dent is assumed to be described by the equation[3]

$$d = k\alpha_d^n \qquad \text{(Eq. 2)}$$

where k and n are constants which may be determined from the log–log plot. Figures 6 and 7 show typical examples of the two plots used to determine k and n. Using Eq. 2, a simple equation for the volume is obtained by assuming circular symmetry—*viz.*,

$$V = \int \pi\alpha_d^2\, \delta d = \int_0^a \pi nk\alpha_d^{n+1}\, \delta\alpha_d$$

$$= \frac{n\pi k}{n+2} a^{n+2} \qquad \text{(Eq. 3)}$$

where a is the maximum value of α_d. The value of a used in Eq. 3 is the same value used later in Eq. 12; n and k were obtained by an iteration procedure with a computer to yield a least-squares fit to the data. In Fig. 7, a line of slope 2 is shown to permit a comparison of the data to a spherical shape.

A Simple Theoretical Model—Tabor's (7) equations for the impact–rebound process are based on a simple model. To the authors' knowledge, this theory has not been discussed previously in the pharmaceutical literature. Therefore, a brief treatment of the concepts should be useful for clarifying the assumptions made and the deviations of experimental values from the calculated ones.

The rebound process is a result of elastic deformation only. It is equal to the reverse process of pushing the sphere back into the dent to produce the same contact area, assumed to be the contact area that has a chordal radius of a. Hertz' equation for the elastic deformation of a sphere and semiinfinite body of homogeneous, isotropic solids is used. Three elasticity relationships are useful—*viz.*,

$$z = \alpha^2 \left(\frac{1}{r_1} - \frac{1}{r_2}\right) \qquad \text{(Eq. 4)}$$

$$F_\alpha \propto z^{3/2} \qquad \text{(Eq. 5)}$$

and

$$\frac{1}{r_1} - \frac{1}{r_2} = \frac{3}{4}\frac{F}{a^3} f(E) \qquad \text{(Eq. 6)}$$

[3] For a spherical indentation of radius r, when $d^2 \ll \alpha^2$, $k \approx 1/2r$, and $n = 2$. Note that $n = 2$ also is a parabola and as long as $d^2 \ll \alpha^2$, the volumes of the two configurations may be considered equal.

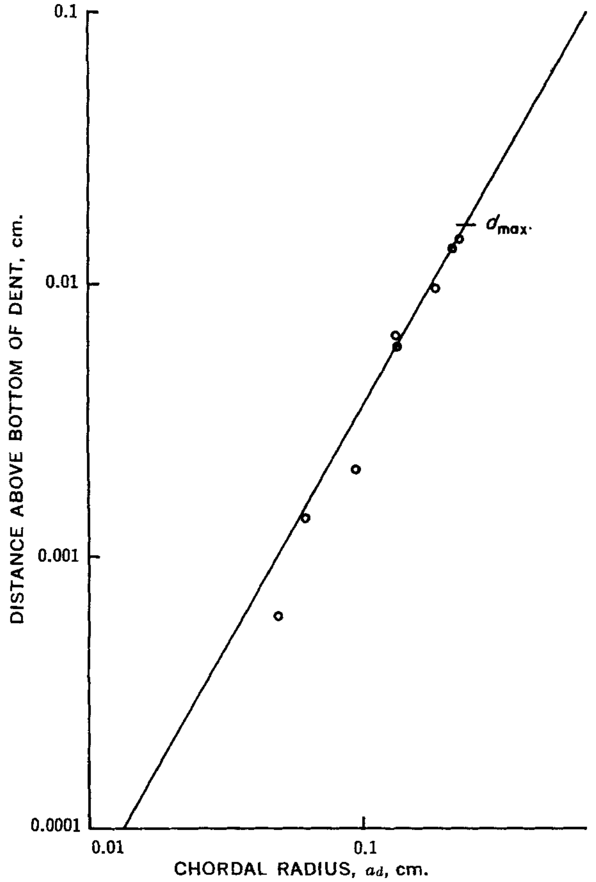

Figure 7—*The same data are used here as in Fig. 6 but the origin has been transposed to the bottom of the dent. The* k *and* n *for Eq. 2 are obtained from a computer analysis which gives the line of this plot;* $d_{max.}$ *is the maximum value of* d *based on Fig. 6.*

where z is the approach distance (elastic penetration) of the sphere into the dent, F_α is the force at penetration z, α is the chordal radius of the contact area at penetration z, r_1 and r_2 are the radii of curvature of the two surfaces before elastic deformation, and

$$f(E) = \frac{1 - \sigma_1^2}{E_1} + \frac{1 - \sigma_2^2}{E_2} \qquad \text{(Eq. 7)}$$

where σ is Poisson's ratio and E is the modulus of elasticity. Subscripts 1 and 2 refer to the spherical indenter and to the permanently dented surface, respectively. Equations 4 and 5 may be used to show that the energy of elastic deformation, E_e, required to push the sphere into the dent to produce contact over a circle of radius a is given by

$$E_e = \int F_\alpha \, \delta z = {}^2/_5 P\pi a^4 \left(\frac{1}{r_1} - \frac{1}{r_2}\right) \qquad \text{(Eq. 8)}$$

where the force at maximum penetration, F, is equal to $P\pi a^2$. Combining Eqs. 6 and 8 gives

$$E_e = {}^3/_{10} P^2 \pi^2 a^3 f(E) \qquad \text{(Eq. 9)}$$

Note that Eq. 9 depends only on the elastic deformation, as does the rebound process. Since $f(E)$ is not known, a complete check of Eq. 9 is not readily made. However, if P (here P is the pressure at maximum penetration) is a constant for different values of a, a plot of log E_e/mg *versus* log a should have a slope of 3. Figure 8 shows such a plot. For most of the examples, the correlation is good. Therefore, it is assumed that Eqs. 8 and 9 describe the rebound process satisfactorily and that P is essentially a constant.

Using Eq. 3, one may show that when the permanent dent is a spherical dent with the depth much less than a, it will have a volume, V_p, equal to $\pi a^4/4r_2$. This may be used to eliminate r_2 in Eq. 8. If it is also assumed that the P in Eq. 1 is equal to the P in Eq. 8, one may combine these equations and solve for P to obtain

$$P = \frac{4r_1}{\pi a^4}(E_p + {}^5/_8 E_e) \qquad \text{(Eq. 10)}$$

or

$$P = \frac{4mgr_1}{\pi a^4}(h_i - {}^3/_8 h_r) \qquad \text{(Eq. 11)}$$

where m is the mass of the sphere, g is the gravitational acceleration constant, h_i is the initial height of the sphere, and h_r is the rebound height. Equation 11 assumes no losses of energy other than the pressure volume work of the permanent deformation. Since $PV_p = mg(h_i - h_r)$, one may divide this by Eq. 11 to obtain V_p:

$$V_p = \frac{\pi a^4}{4r_1}\left(\frac{h_i - h_r}{h_i - {}^3/_8 h_r}\right) \qquad \text{(Eq. 12)}$$

Figure 9 is a plot of the volumes calculated using Eq. 12 *versus* the "photo" volumes, *i.e.*, the volumes estimated from photographs such as Fig. 5. Equation 12 yields slightly larger values than the photo volumes. Obviously, both values may be in error. Errors in the photo volumes are expected to have random distribution, but undetected systematic errors are possible. These could lead to an underestimate of the dent volumes. However, Eq. 12 estimates may be expected to be slightly larger than the true volumes because frictional, accoustical, and hysteresis losses have been neglected. Also the mean deformation pressure may not be a constant during the impact because of the dynamic nature of the process. The last assumes P is larger when the sphere is moving at a larger velocity and goes to a minimum as the sphere velocity goes to zero[4]. This too would result in an overestimate of the volume by Eq. 12. However,

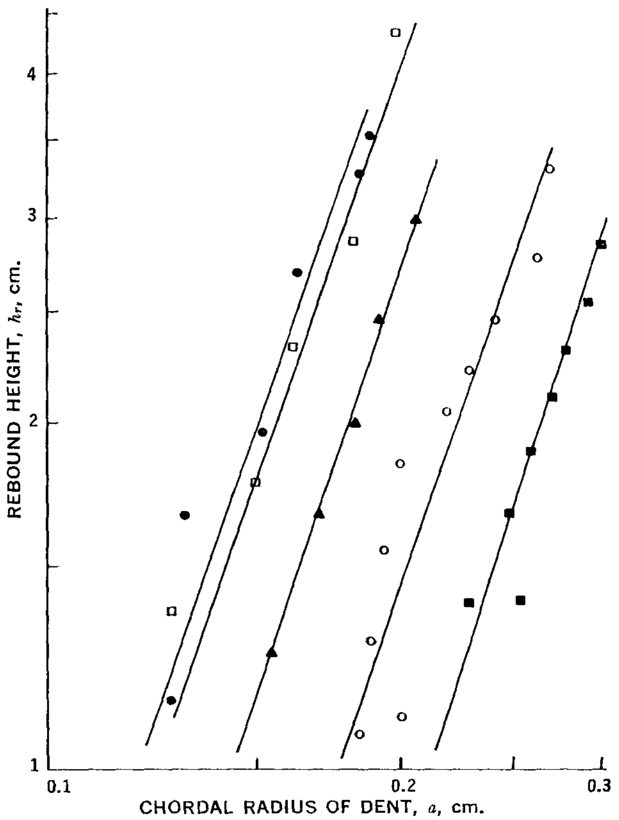

Figure 8—*Comparison of Eq. 9 with the experimental data. Solid lines have theoretical slope 3. Key:* ●, *spray-dried lactose, Lot B,* $\rho_r = 0.749$; □, *sitosterol,* $\rho_r = 0.868$; ▲, *sucrose,* $\rho_r = 0.818$; ○, *spray-dried lactose, Lot A,* $\rho_r = 0.690$; *and* ■, *a tablet formulation,* $\rho_r = 0.574$.

[4] Even so, P in Eq. 9 may be constant because it is concerned only with P at zero velocity of the sphere.

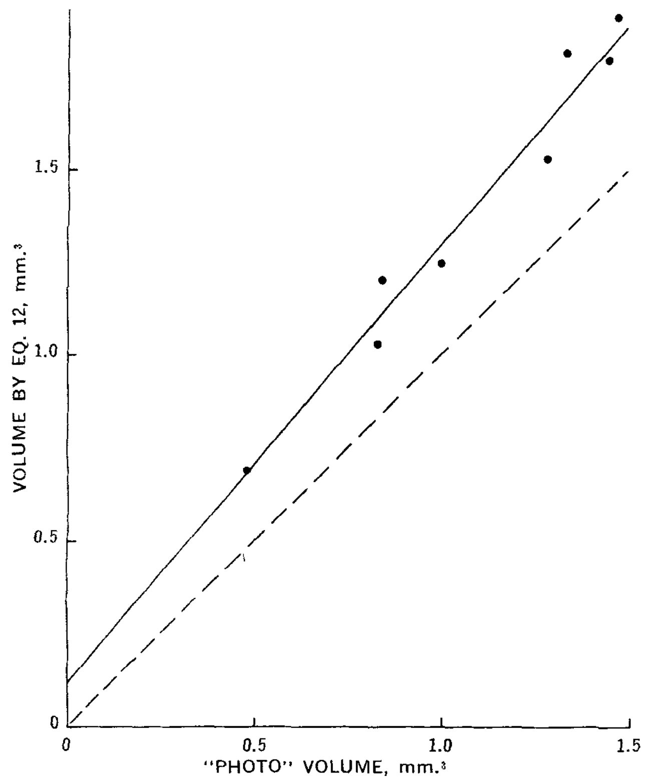

Figure 9—*Comparison of the values obtained by two methods of estimating the volumes of the permanent indentation. Values based on Eq. 12 are somewhat larger than those based on the photographs.*

consolidation beneath the dent during penetration could cause P to increase. This effect should be detected by a more rapid increase in a than for a slope of 3 in Fig. 8. Only one portion of one of the plots shows significant deviation in this direction. Therefore, this effect appears generally to be insignificant.

Based on these studies, the authors conclude that probably Eq. 12 overestimates V_p by a small amount and that the companion Eq. 11 therefore would underestimate the mean pressure of plastic deformation. However, these small deviations from the true values are not expected to be significant impediments to the application of this technique of hardness testing.

Figure 10 shows a plot of log P *versus* ρ_r, where ρ_r is the relative density of the compact, *i.e.*, the bulk density divided by the absolute density of the crystalline materials. Extrapolations to $\rho_r = 1$ may be questionable but are considered useful estimates of the value of P for the crystalline solid.

Additional Experimental Results—The area over which molecular forces may act after two solids have been pressed together depends on the effectiveness of the relieved elastic stresses in producing separation of the surfaces. For the geometric model considered here, this "contact area" is a function of the ratio of r_2/r_1. If, in deriving Eq. 10, r_2 had not been eliminated, one could have obtained

$$r_2/r_1 = 1 + \frac{5}{8}\frac{E_e}{E_p} \qquad \text{(Eq. 13)}$$

Rearranging Eq. 6 and replacing a^3 with $(F/p\pi)^{3/2}$, one notes that

$$\frac{r_1}{r_2} = 1 - \frac{3}{4}\frac{r_1 P^{3/2}\pi^{3/2}}{F^{1/2}} f(E) \qquad \text{(Eq. 14)}$$

Obviously, E_e/E_p is not a constant independent of the applied force F since r_1/r_2 is a function of F. Combining the above with Eq. 13 yields

$$\frac{E_p}{E_e} = \frac{5}{6}\frac{F^{1/2}}{\pi^{3/2} r_1 P^{3/2} f(E)} - \frac{5}{8} \qquad \text{(Eq. 15)}$$

Since $F^{1/2} = (P\pi)^{1/2}a$ when P is constant, this relationship may be tested in a manner analogous to the test of Eq. 9. A plot of E_p/E_e *versus* a is shown in Fig. 11. The lines in Fig. 11 were drawn through the $-5/8$ intercept. Equation 15 contains both the plastic and elastic parts of the impact process. Since this test is independent of the photo method and any associated errors in it, the results shown in Fig. 11 strongly reinforce the argument that the model and the equations provide good estimates of the yield pressure.

The following extension of these concepts to the interaction between particles was reported previously (11). If one considers a sphere of radius r_1 at rest in a spherical dent of radius r_2, the separation, S, of the two spherical surfaces at a chordal radius, α, from the point of tangency is given for $\alpha \ll r_1$ by

$$S = \frac{\alpha^2}{2}\left(\frac{1}{r_1} - \frac{1}{r_2}\right) \qquad \text{(Eq. 16)}$$

Using Eq. 13 to eliminate r_2 and rearranging give

$$\alpha^2 = 2r_1 S\left(1 + \frac{8E_p}{5E_e}\right) \qquad \text{(Eq. 17)}$$

If it is assumed that the attraction resulting from intermolecular forces is effective over an area $A_c = \pi a_c^2$ at which point $\alpha = a_c$ and S equals S_m, the maximum separation effective in bonding, then one obtains

$$A_c = \pi a_c^2 = 2\pi r_1 S_m\left(1 + \frac{8E_p}{5E_e}\right) \qquad \text{(Eq. 18)}$$

Combining this with Eq. 15 yields, for a single contact point,

$$A_c = \frac{8S_m F^{1/2}}{3\pi^{1/2}P^{3/2}f(E)} \qquad \text{(Eq. 19)}$$

This equation predicts that A_c should increase as the $1/2$-power of the force applied and decrease as the $3/2$-power of the plastic yield value of the yielding solid.

If one extends this to the application of a total force, F_t, on a given cross-sectional area of a powder bed of monodispersed particles, and if one assumes that only one of the particles deforms so that after elastic rebound one of each pair of contacting particles retains a radius of r_1, it is obvious that the total effective contact area will be

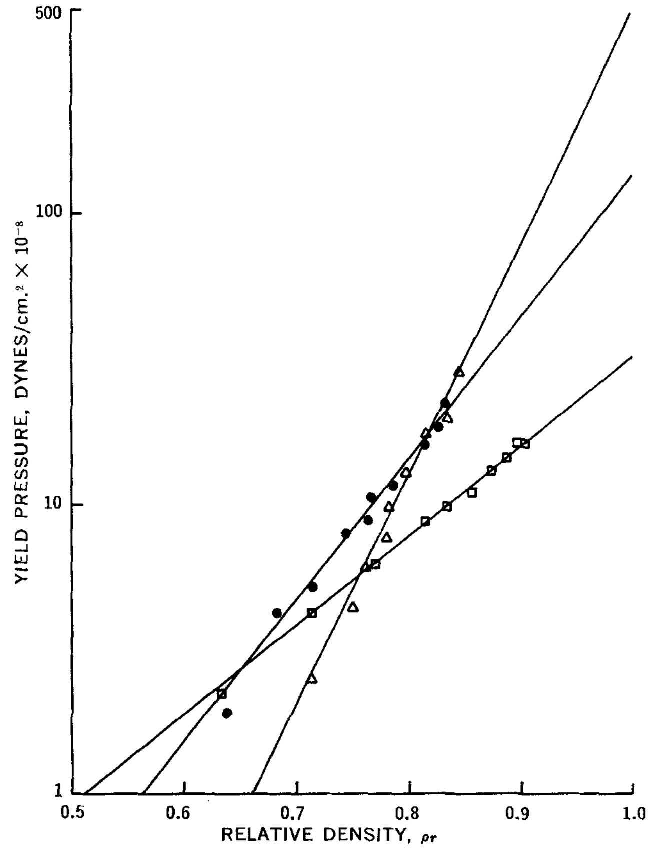

Figure 10—*Mean yield pressure appears to follow a logarithmic relationship with the bulk density of the compact. Key:* ●, *spray-dried lactose, Lot A;* △, *sucrose; and* □, *tablet formulation.*

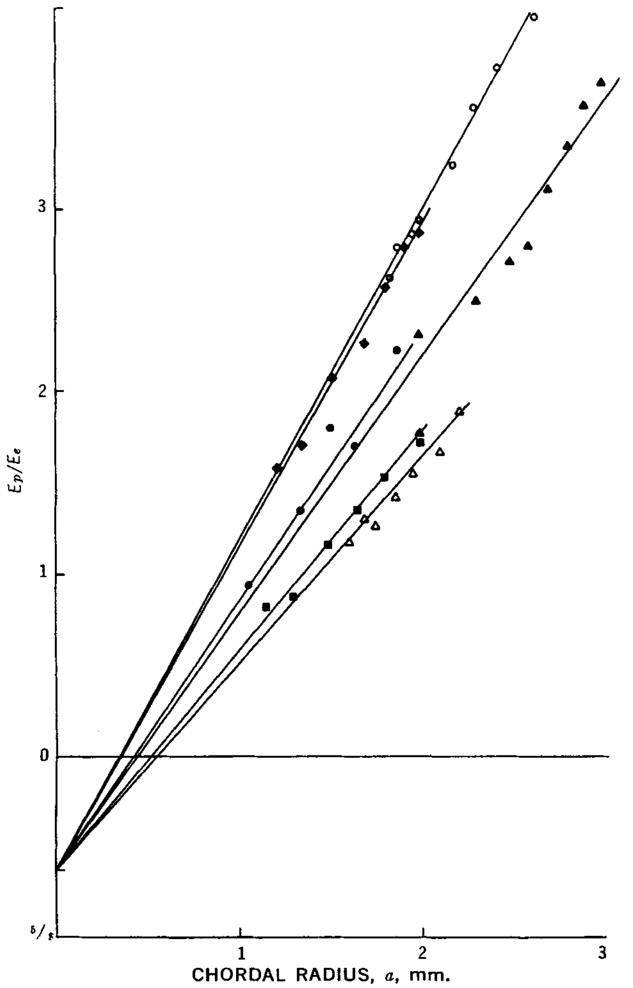

Figure 11—*Comparison of the experimental data with Eq. 15. The lines are drawn through the theoretical value of the intercept, $-^5/_8$. Key:* ○, *spray-dried lactose, Lot A, $\rho_r = 0.690$;* ●, *spray-dried lactose, Lot B, $\rho_r = 0.749$;* ◆, *sucrose, $\rho_r = 0.818$;* ▲, *tablet formulation, $\rho_r = 0.574$;* △, *tablet formulation, $\rho_r = 0.752$; and* ■, *sitosterol, $\rho_r = 0.868$.*

N_cA_c, where N_c is the number of contacts. If the packing remains the same, $N_c \propto 1/r_1^2$ and $F \propto r_1^2$. Therefore, $F_t = N_cA_c \propto 1/r_1$. Qualitatively, this conforms with experience, *i.e.*, smaller particles form more cohesive powder beds. According to Krupp (12), with small particles, attractive forces may deform contacting surfaces without externally applied loads. This effect must be added to the relationship that considers only the role of mechanical properties.

DISCUSSION

The relationship between the mean pressure of deformation and the yield stress is not simple, especially in a heterogeneous, porous compact. Friction and other factors influence this relationship. In the absence of friction, the mean pressure is equal to approximately three times the yield stress of the solid (7). Since the exact relationship is unknown in the real case, it is probably better to avoid any statement on the yield stress. The mean pressure required to produce the indentation may be used as an indication of the hardness, *i.e.*, the resistance to permanent deformation.

Obviously, pressures as large as those shown in Fig. 10 can only be developed by the rapid deceleration of the sphere. Tabor (7) derived Eq. 20 to estimate the time, *t*, after contact required to bring the velocity of the sphere to zero:

$$t = \frac{\pi}{2}\sqrt{\frac{m}{2\pi P r_1}} \qquad \text{(Eq. 20)}$$

Using this equation, values in the range of 10^{-4} sec. are obtained. The time of contact is not dependent on the velocity of the sphere unless *P* is a function of velocity. *P* has been assumed constant in all the equations adopted from Tabor's book (7). The time of impact is much less than the time of compression in a rotary tablet machine.

Since the impact–rebound technique permits the separation of the elastic from the anelastic displacement and energy, it may be feasible to characterize the elastic properties also. This aspect is being explored in the authors' laboratory.

The assumptions made in the use of this technique may cause one to question the validity of the method. Certainly, not all energy losses are due to plastic deformation. However, some workers (13) reported coefficients of restitution greater than 0.98 when hard materials were used with small impact velocities to avoid permanent deformation. Therefore, it is concluded that the basic concepts are essentially correct.

CONCLUSION

The use of an impact arrangement permits the energy and volume of the permanent deformation to be determined. The average deformation pressure is calculated from the same data. Equations used for metallurgical specimens have been shown to have applicability in the characterization of compacts of organic solids.

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ACKNOWLEDGMENTS AND ADDRESSES

Received June 17, 1970, from the *Pharmacy Research Unit, The Upjohn Company, Kalamazoo, MI 49001*

Accepted for publication January 6, 1971.

The authors are grateful to Mr. B. D. Haffner and Mr. J. W. Swanson for contributions to the design of the apparatus and for making some of the measurements. The basic design was the work of Mr. C. Sperry.