method of film application may lead to differences in the steady-state accumulation of moisture at the distal surface. This, in turn, would alter the film's permeation characteristics through plasticization. The data in Fig. 2 suggest that the more intimate the contact between the film and the tablet and/or disk, the less the degree of moisture accumulation and the lower the plasticizing effect.

A second important factor relating to moisture uptake by films coated on disks or tablets is seen in Fig. 2. When contrasting the plot for the film sprayed onto the disk made with the press (B) with that for the tablet (D) made on the single-punch tablet press, it is apparent that the energy of activation varies with the character of the material being coated. Thus, the disks were prepared under approximately 13,000 psi pressure while the tablets were formed under 35,000 psi pressure. This difference is further emphasized in Table II, which gives the rate of water vapor uptake by the uncoated disks and tablets. The rate of water vapor uptake is approximately 20–50 times faster and obviously more temperature dependent for the disks than for the tablets.

Over the pressure range studied, it is reasonable to expect that the disks would have a higher porosity and specific surface than the tablets. This would account for the more rapid uptake of moisture by the disks. Assuming that the specific surface and porosity of the compressed tablet are critical and, hence, rate limiting, one would expect the moisture uptake by the disks to become related increasingly to the temperature-dependent rate of water vapor condensation. When a film is applied, however, the situation changes. The rate of water absorption by the disks, which in the uncoated state are able to absorb water considerably faster at all temperatures, is largely controlled by the film. Thus the activation energy for the spray-coated disks (B) is the same as that for the free films (C).

The coated tablets, however, present two barriers to the water permeation and absorption process. The first is due to the film. The second is the relatively low specific surface and low porosity interface of the compressed tablet. In comparison to the disk, the passage of water molecules into the tablet is restricted and the activation energy becomes a function of both factors. In support of this argument, the energy of activation for the water vapor absorption process with coated tablets is 11.9 kcal/mole, in reasonable agreement with the sum of the activation energies for permeation of the film (5.3 kcal/mole) and the rate of water uptake by the uncoated tablet (5.5 kcal/mole).

In conclusion, the results of this study indicate that the rate of water vapor absorption by solid dosage forms, film coated with a hydrophilic powder, is determined by the method of film application and the physical characteristics of the coated material. The data suggest that, with this type of film, the rate of absorption may be decreased by ensuring that the film is in intimate contact with the dosage form and by compressing the tablet to the maximum degree feasible compatible with disintegration considerations.

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Flexure Test for Determination of Tablet Tensile Strength

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Abstract □ A new method for determining the tensile strength of tablets is proposed utilizing two fulcrums and a knife edge. The fulcrum and knife edge pieces were affixed to the platens of a motorized tablet hardness tester. The new method was found to give rapid, reproducible results which agreed well with tensile strength values obtained from diametral compression. In contradistinction to diametral compression, tablets invariably failed in tension using the new method, as evidenced by uniform splitting into halves.

Keyphrases □ Tensile strength, tablets—new flexure test, apparatus and equations □ Tablets, tensile strength—new flexure test for determination, apparatus and equations □ Flexure test—determination of tablet tensile strength, compared to diametral compression method, apparatus and equations

Several approaches have been utilized in the evaluation of tablet strength. Some of the more popular designations for values relating to the strength of tablets are bending resistance (1), crushing force (2), and tensile strength (3–7). Of these, tensile strength is of more fundamental importance because it is independent of tablet dimensions and provides a measure of the inherent strength of the compacted material (6, 7).

DISCUSSION

Fell and Newton (3, 4) determined tensile strength using a diametral compression test similar to that commonly used to determine tablet crushing strength where tablets are compressed between two flat platens. If the tablets fail in tension, tensile strength may be computed from:

$$\sigma_0 = \frac{2P}{\pi D t} \qquad \text{(Eq. 1)}$$

where σ_0 is the tensile strength, P is the applied load, D is the tablet diameter, and t is the tablet thickness. Based on elastic theory, the derivation of this equation may be found in standard texts (8–10). In general, this involves a consideration of radial pressures and concentrated loads acting along a diameter of a circular disk and an analysis of the state of stress in the circular disk in terms of rectangular stress components. The principal assumptions are that the disk obeys Hooke's law, that the disk is isotropic, and that the modulus of elasticity in tension and compression is the same. For the vertical central section, which would represent the section in a flat-faced tablet where failure takes place, compressive stresses are at a minimum at the center of the load diameter and infinite at the extremities where the

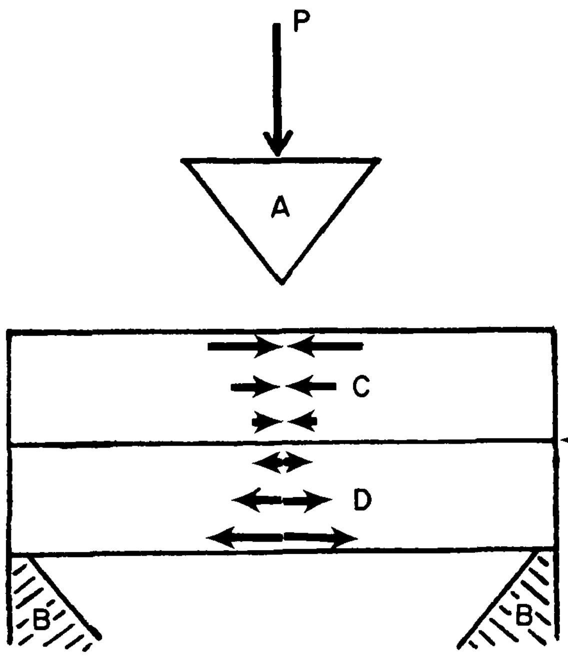

Figure 1—*Schematic drawing showing a vertical, central section of a tablet. Key: A, straight edge; B, fulcrums; C, compression; D, tension; E, neutral plane; and P, load.*

load is being applied, and a uniform tensile stress exists along the load diameter. Under the theoretical conditions of ideal line loading, these high compressive stresses and resulting high shear stresses prevent the initiation of failure in tension. In actual practice, however, failure in tension is possible because the load is distributed over an actual area of contact, thereby reducing the magnitude of the compressive and shear stresses at the extremities of the load diameter.

An alternative approach, proposed here, to the measurement of tensile strength in the plane described by Fell and Newton is to fracture the tablet by means of a straight edge in a flexure or bending test. The tablet is supported at two extreme ends of one of its flat faces and a knife edge, extending the width of the tablet on the opposite face (Fig. 1) is caused to exert an increasing load on the center of the tablet until it fractures. When a load is applied on the top face at the center and uniformly through the diameter of the tablet, the vertical, central plane feels both compression and tension (Fig. 1). The top half experiences compression while the lower half experiences tension. Therefore, an imaginary plane (E) can be drawn through the center of the tablet parallel to the tablet faces which feels neither compression nor tension. This is called the neutral plane. The neutral axis (N, Fig. 2), located at the center of the neutral plane, coincides with the tablet diameter and is parallel to the knife edge applied to the top tablet face. Compression and tension in the vertical cross section, in which the neutral axis is at the center, are assumed equal in magnitude but of opposite signs. The other main assumptions are the same as noted before for the diametral compression treatment.

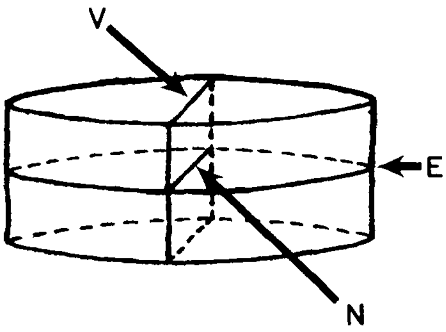

Figure 2—*Schematic drawing showing vertical, central plane and neutral axis. Key: V, vertical, central plane; E, neutral plane; and N, neutral axis.*

The general form of the flexure equation, from elastic theory (9, 10), is as follows:

$$\sigma = My/I \qquad \text{(Eq. 2)}$$

where σ is bending stress, M is the bending moment, y is the distance from the neutral axis to any point or element of area in the vertical central section, and I is the moment of inertia. For a flat-faced tablet, the vertical cross section normal to the neutral plane is rectangular (unless tablet thickness is equal to tablet width and a square is generated), and its moment of inertia is given by:

$$I = \frac{bh^3}{12} \qquad \text{(Eq. 3)}$$

where I is the moment of inertia, b is tablet width (diameter), and h is tablet thickness. Since, for these purposes only, the half of the tablet that experiences tension is of interest:

$$y = \frac{h}{2} \qquad \text{(Eq. 4)}$$

By definition, maximum bending moment is:

$$M = \frac{Fb'}{4} \qquad \text{(Eq. 5)}$$

where F is the load observed when the tablet fractures, and b' is the distance from fulcrum to fulcrum. Making these substitutions in Eq. 2 results in:

$$\sigma = \frac{3Fb'}{2bh^2} \qquad \text{(Eq. 6)}$$

Since the maximum bending moment is $Fb'/4$, the tablet is loaded by a couple or resisting moment of $Fb'/8$ at each of its ends. Therefore, the equation must be divided by two, yielding:

$$\sigma_0 = \frac{3Fb'}{4bh^2} \qquad \text{(Eq. 7)}$$

where σ_0 is now the tensile strength.

EXPERIMENTAL

Comparison of Tensile Strength Measurement by Two Methods—The primary objective of this study was to compare tablet tensile strength as determined by diametral compression and by flexure. Diametral compression was carried out after the manner of Fell and Newton (3, 4) by compressing tablets between the flat platens of a constant-speed motorized tablet hardness tester[1]. Tensile strength was computed from the force required to cause failure using Eq. 1. Only the data from tablets that had obviously failed in tension, as evidenced by splitting into halves along the load diameter (11), were accepted.

For the flexure test, a knife edge and fulcrum piece machined from tool steel were affixed to the platens of the hardness tester (Fig. 3). Care was taken to align the two pieces so that the knife edge would contact the test tablets at their diameters, and the knife edge was of sufficient length to act across the entire diameter of the test tablets. The fulcrum piece was machined to an overall height and breadth equal to the diameter of the test tablets to facilitate the proper alignment of the tablets against the fulcrum piece. In this case, tensile strength was computed from the force required to cause failure using Eq. 7. The principal dimensions of the knife edge and fulcrum pieces are given in Fig. 4.

Three direct compression tablet fillers representative of different types were used. They were lactose[2] USP, compressible starch[3], and microcrystalline cellulose[4] NF. No active ingredients were included. No additives other than a lubricant were included and then only in the case of lactose, since the compressible starch and microcrystalline cellulose are known to be self-lubricating when used alone (12, 13). Lactose was lubricated with 0.75% mag-

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[1] Heberlein model 2E/106, series 7203, Key Industries, Farmingdale, N.Y.

[2] Fast-Flo lactose, Foremost Dairies Inc., San Francisco, Calif.

[3] Sta-Rx 1500 starch, A. E. Staley Manufacturing Co., Decatur, Ill.

[4] Avicel PH 102, F. M. C. Corp., American Viscose Division, Newark, Del.

Table I—Comparison of Values for Tensile Strength as Determined by Flexure Test and Diametral Compression

Filler	Compression Force, kg	Tablet Dimensions, cm		Fracturing Force, kg ± SD[a]		Tensile Strength, kg/cm² ± SD[a]	t Statistic[b]
		Thickness	Diameter	Diametral Compression	Flexure Test		
Microcrystalline cellulose	208	0.201	1.118	11.8 ± 0.5	—	33.4 ± 1.4	
	208	0.201	1.118	—	2.0 ± 0	33.2 ± 0	0.428[c]
	212	0.245	1.118	14.7 ± 0.3	—	34.2 ± 0.7	
	212	0.244	1.118	—	3.1 ± 0.2	35.0 ± 2.2	1.04[c]
	254	0.214	1.118	15.3 ± 0.6	—	40.7 ± 1.6	
	254	0.213	1.118	—	2.8 ± 0.2	41.5 ± 1.1	1.10[c]
	269	0.240	1.118	18.4 ± 0.4	—	43.7 ± 0.9	
	269	0.242	1.118	—	3.7 ± 0.1	42.4 ± 1.1	2.75[d]
	316	0.190	1.118	17.8 ± 0.5	—	53.4 ± 3.5	
	316	0.190	1.118	—	2.6 ± 0.3	48.3 ± 5.6	2.23[d]
Compressible starch	1776	0.354	1.118	13.9 ± 1.3	—	22.4 ± 2.1	
	1776	0.353	1.118	—	3.7 ± 0.3	20.0 ± 1.9	2.55[d]
Lactose	810	0.304	1.118	14.4 ± 0.7	—	27.0 ± 1.3	
	810	0.303	1.118	—	3.8 ± 0.3	27.8 ± 2.2	0.940[c]

[a] Standard deviation, based on 10 determinations. [b] Student's t test for difference comparing the value for tensile strength determined by the flexure test to that determined by diametral compression for each run. [c] Difference not significant at 5% level. [d] Difference not significant at 1% level.

nesium stearate[5] USP which had been previously passed through an 80-mesh sieve to facilitate homogeneous blending.

The lubricated blend (500-g batch) was prepared by mixing[6] for 12 min, with the intensifier bar running for only the final 2 min. Tablets were prepared on a rotary tablet press[7] which had been instrumented with resistance strain gauges as previously described (14, 15) to monitor compression force. Press speed was 25 rpm, and all tableting was conducted in a room where temperature and relative humidity were maintained at 26 ± 1° and 35 ± 5%, respectively. Only a single station was run in order to help minimize tooling errors, and the tooling consisted of circular, flat-faced, 1.111-cm punches and die. Approximately 250-g quantities of powder were added to the hopper for each run, and tablet samples were not taken until after the press had run at least 2 min. Tensile strengths by the two methods were compared at a single compression force and tablet thickness in the cases of compressible starch and lactose. For microcrystalline cellulose, comparisons were made at five different compression forces and thicknesses because this filler makes strong tablets over a wide range of compressional force. After each tableting run, the tablets were measured for thickness and width using an inch measure[8]. Ten replicate tensile strength determinations were made using each method.

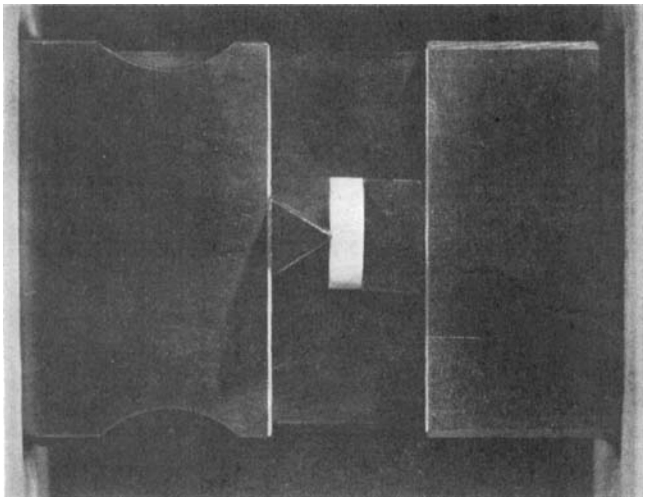

Figure 3—*Arrangement of knife edge, fulcrum piece, and tablet between the platens of the hardness tester.*

Effect of Tablet Thickness—In this study, the effect of tablet thickness on bending force and tensile strength was examined. Microcrystalline cellulose was selected for its high compressibility. Because of the very thick tablets generated, 0.3% magnesium stearate was added as a lubricant, even though, as noted previously, this filler does not normally require a lubricant when used alone. Blending and tableting were carried out as previously described. Five batches of tablets, each representing a different tablet thickness, were prepared with the same compression force of 604 ± 15 kg by changing both the die-fill and pressure settings for each run. Ten tablets from each batch were measured for thickness and diameter and fractured in the flexure test in the manner previously described.

RESULTS

From Table I it is clear that there is very good agreement in all cases between the two methods of tensile strength measurement. Application of the Student t test revealed that the differences in tensile strength were not significant at the 1% level in all comparisons. In four of the seven comparisons, the differences were not significant at the 5% level. Furthermore, the standard deviations for either method compare well with the variances previously reported for diametral compression (4).

The constants in Eq. 7 may be collected, and the equation may be rearranged to yield:

$$F = K\sigma_0 h^2 \qquad \text{(Eq. 8)}$$

where $K = 4b/3b'$. Thus, a plot of F against h^2 should yield a straight line passing through the origin with a slope = $K\sigma_0$, provided that tablet compression force and the force lost to the die wall are the same for the tablets at each thickness. As can be seen in Fig. 5, a straight line was produced when F was plotted against h^2 for various thicknesses of microcrystalline cellulose tablets compressed at the same compression force. The experimental data were fitted by a linear regression, least-squares plot, and the experimentally determined intercept of −0.1 kg/cm is in close agreement with the theoretically predicted intercept of zero. It is likely that the inclusion of magnesium stearate in these batches helped minimize any differences in the force lost to the die wall at the tablet thicknesses studied. However, as would be expected, the inclusion of magnesium stearate also materially weakened these tablets. From the slope of the curve, the tensile strength of these tablets was found to be 29.8 kg/cm². Comparison with Table I reveals that this figure is lower than any value found for the unlubricated microcrystalline cellulose tablets, even though they were compressed with only about one-third to one-half the compression force used to compress the lubricated batches.

In general, the flexure test was found to be a rapid, reproducible method for determining tablet tensile strength. The tensile strengths of very strong tablets can be measured more readily by this method than by diametral compression since the fracturing

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[5] Ruger Chemical Co., New York, N.Y.
[6] Patterson Kelly V-mixer.
[7] Stokes model RB-2.
[8] Federal model C815, Federal Products Corp., Wayne, Pa.

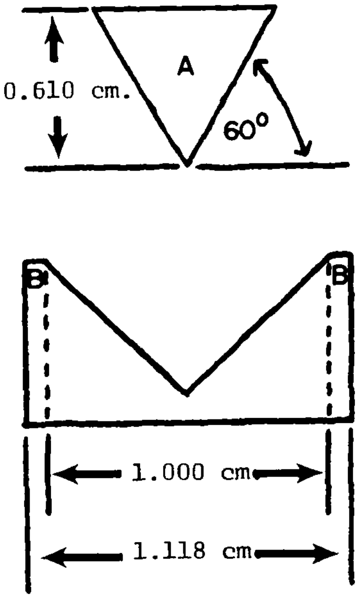

Figure 4—*Principal dimensions of straight edge and fulcrum setup. Key: A, straight edge; and B, fulcrums.*

forces obtained by the bending method were always less than those observed for fracture by diametral compression for the distance between fulcrums employed. However, the flexure test measurement has a higher coefficient of variation. If tablets are of exceptionally low strength, it may be necessary to reduce the distance between the fulcrums to get measurable fracturing forces. Tablets invariably failed in tension in the flexure test, as evidenced by uniform splitting into halves. Diametral loading can shatter the tablet and there can be noticeable shearing at the extremes of the vertical cross section where loading takes place rather than perfect diametrical splitting in one plane. To minimize this problem of the diametral compression test, Fell and Newton (4) recommended the use of soft padding material between the tablet and the platens. Also, Newton (16) noted that diametral compression does not work well if the tablets are not flat faced. The flexure test should be readily applicable to convex tablets if the moment of inertia of the vertical, central plane involved in tablet fracture is known. This is presently being studied in these laboratories.

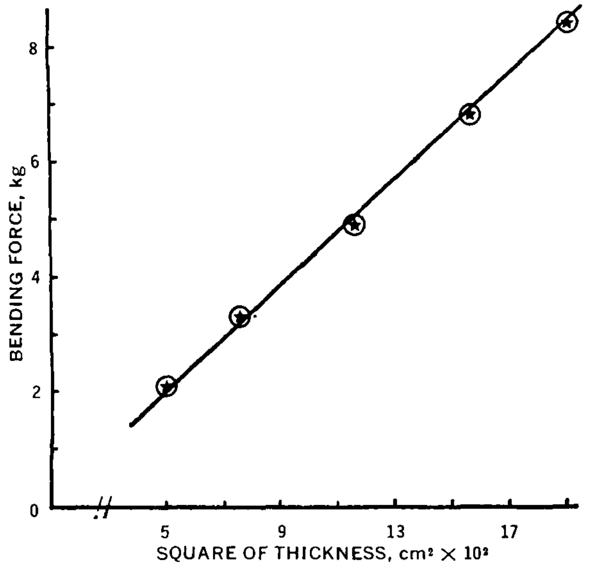

Figure 5—*Plot of bending force versus square of thickness for microcrystalline cellulose tablets containing 0.5% magnesium stearate. Slope = 44.3, and tensile strength = 29.8* kg/cm^2.

SUMMARY AND CONCLUSIONS

A flexure test for determining the tensile strength of tablets was proposed wherein tablets are compressed between two fulcrums and a knife edge. The fulcrum and knife edge pieces were affixed to the platens of a motorized tablet hardness tester. The method was rapid and the tablets invariably failed in tension, as evidenced by uniform splitting into halves. Experimental results with three different fillers (microcrystalline cellulose, lactose, and compressible starch) showed excellent agreement with tensile strength values obtained by diametral compression. In another study with microcrystalline cellulose, tensile strength was found to be independent of tablet thickness, as predicted by theory. For the distance between fulcrums employed, the fracture force in flexure was less than that required in diametral compression, thus making the new method more suitable for very strong tablets than diametral compression. For very weak tablets, it may be necessary to reduce the distance between fulcrums to get measurable fracture forces.

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