Chem., **285,** 172 (1950).
(8) H. Gershon, R. L. Rodin, R. Parmegiani, and P. K. Godfrey, *Contrib. Boyce Thompson Inst.*, **24,** 245 (1970).
(9) J. Donahue, in "Structural Chemistry and Molecular Biology," A. Rich and N. Davidson, Eds., W. H. Freeman, San Francisco, Calif., 1968, pp. 443–465.
(10) R. H. Baechler, *Proc. Am. Wood Preserv. Assoc.*, **1939,** 364.
(11) H. Engelhard, O. Müller, and K. Bertl, *Zentralbl. Bakteriol. Parasitenkd., Abt. I*, **153,** 326 (1949).
(12) R. W. Finholt, M. Weeks, and C. Hathaway, *Ind. Eng. Chem.*, **44,** 101 (1952).
(13) F. L. M. Pattison, W. C. Howell, A. J. McNamara, J. C. Schneider, and J. W. Walker, *J. Org. Chem.*, **21,** 739 (1956).
(14) S. Voerman and A. K. Minks, *Environ. Entomol.*, **2,** 751 (1973).
(15) G. M. Bennett and H. Gudgeon, *J. Chem. Soc.*, **1938,** 1679.
(16) H. Gershon and L. Shanks, *Can. J. Microbiol.*, **21,** 1317 (1975).
(17) H. Gershon and R. Parmegiani, *Appl. Microbiol.*, **10,** 348 (1962).
(18) H. Gershon, R. Parmegiani, and W. J. Nickerson, *ibid.*, **10,** 556 (1962).
(19) C. Hansch and E. J. Lien, *J. Med. Chem.*, **14,** 653 (1971).

New Instrumentation for Determining Flexure Breaking Strength of Capsule-Shaped Tablets

GERALD GOLD ˣ, RONALD N. DUVALL, and BLAZE T. PALERMO

Received June 14, 1978, from the *Applied Pharmaceutics Laboratory, Miles Laboratories, Inc., Elkhart, IN 46514.* Accepted for publication October 22, 1979.

Abstract □ New instrumentation to measure the flexure breaking strength of capsule-shaped tablets was developed. It consisted of a mechanical linkage to apply the breaking load at a uniform rate and a strain-gauge instrumented cantilever beam to convert the load to a proportional millivolt response on a recorder. A recorder tracing was obtained of increasing load with time, with a break in the tracing denoting the tablet's flexure breaking strength. Measurements were made for different tablet hardnesses, and a plot of tablet hardness *versus* flexure breaking strength yielded a profile of the mechanical strength of the tablet. The instrumentation was shown to have application for determining the effects of tablet thicknesses, tablet ingredients, tablet sizes, cup depths, and bisect dimensions on tablet strength.

Keyphrases □ Flexure breaking strength—determination for capsule-shaped tablets, new instrumentation □ Tablet breakage—capsule-shaped tablets, determination of flexure breaking strength, new instrumentation □ Hardness, tablets—flexure breaking strength, determination for capsule-shaped tablets, new instrumentation

Although tablet breakage is a problem often encountered in the manufacture, packaging, and distribution of capsule-shaped tablets, little information is available on the subject. Several factors that need to be studied for their effects on breakage are the ingredients, size, shape, and dimensions of the tablets. To evaluate these factors, the flexure breaking strength of the tablet must be determined. At present, there is no generally accepted mechanical method to measure the flexure breaking strength of tablets with sufficient accuracy and precision.

BACKGROUND

A widely used but subjective flexure test employed in practice is to break the tablet between the thumb and fingers and to evaluate both the force necessary to break the tablet and the sharpness of the snap. An instrument constructed to simulate the manual method was described (1) in which a pneumatic tablet hardness tester was modified by the addition of two auxiliary pieces, one attached to the plunger and the other to the anvil. Increasing pressure is applied through the plunger on the center of the tablet face until it breaks to measure the fracture resistance. These investigators found that the fracture resistance was directly proportional to tablet thickness and pointed out the importance of this finding for reducing tablet breakage. They also indicated that additional research was necessary to design an instrument that would be more versatile and accurate.

In another study, a flexure tester was constructed by attaching a knife edge and two fulcrums to the platens of a motorized tablet hardness tester (2). The flexure tester was found to be a rapid, reproducible instrument for determining the tablet tensile strength of very strong tablets but could not be used for very weak tablets.

Two related procedures have been developed to measure resistance to bending. In one procedure, mercury is added to provide the weight to break the tablet (3). In the other procedure, a special tablet form is used that is unrelated to the manufactured product (4). Both procedures, however, are impractical for use in industry.

The objective of this study was to develop new instrumentation that can be calibrated readily for increased accuracy, measure a wide range of tablet strengths, and be of practical use in a product development laboratory. This paper describes the instrument developed and indicates

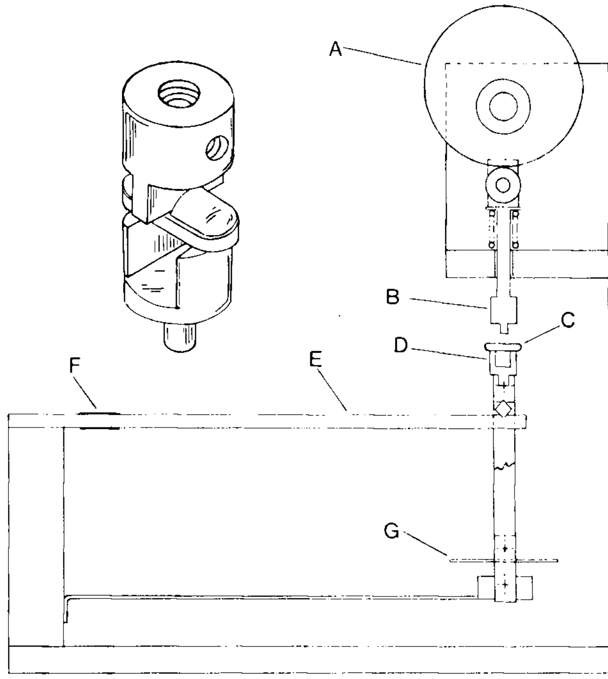

Figure 1—*Instrumentation for measuring flexure breaking strength. Key: A, motor-driven cam; B, plunger; C, capsule-shaped tablet; D, anvil; E, cantilever beam; F, strain gauges; and G, weight platform for calibration.*

0022-3549/ 80/ 0400-0384$01.00/ 0

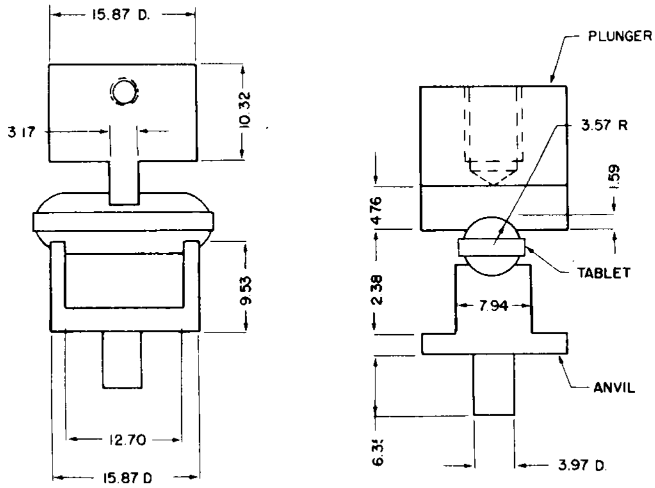

Figure 2—*Dimensions of the plunger and anvil in millimeters.*

how the measurements may be used in product development to evaluate factors affecting tablet breakage.

EXPERIMENTAL

The flexure tester (Fig. 1) was constructed by modifying the plunger tip and anvil of instrumentation described previously for measuring granule strength (5). Dimensions of the modified plunger tip and anvil are given in Fig. 2.

The flexure tester consists of a mechanical linkage for applying the load at a uniform rate and a strain-gauge instrumented cantilever beam and recorder for measuring the magnitude of the applied load. To measure flexure breaking strength, a single capsule-shaped tablet (C) is placed on the anvil (D), and the cam (A) is rotated at a uniform rate. Rotation of the cam at one revolution per 45 sec is controlled by a link chain, gear motor[1], and speed control switch[2].

The plunger (B) presses down across the middle of the tablet and bends the instrumented cantilever beam (E), thereby deforming the strain gauges (F). This deformation results in a millivolt response across the strain-gauge bridge circuit, which is measured on a recorder[3] with a 0–2-mv range for 25.4-cm full scale and a chart speed of 0.33 cm/sec. A tracing is obtained of increasing load with time, and a measurement of the tablet breaking strength is obtained at tablet breakage. The cam makes one complete revolution for each measurement.

The calibration procedure is built into the instrumentation and is performed each time the instrument is operated. A linear relationship exists between the weight on the calibration platform and the millivolt response on the recorder. The instrument is calibrated by adding weights to the calibration platform and adjusting the gain and zero controls to obtain the desired full-scale deflection on the recorder. Full-scale deflection can be varied from 200 g to 10 kg.

A typical recorder tracing of a flexure breaking strength measurement is shown in Fig. 3. The load is applied at a uniform rate by the speed control switch, which regulates the descent rate of the plunger. A break in the tracing of the increasing load with time indicates the tablet flexure breaking strength. In this tracing, the flexure breaking strength is 3 kg.

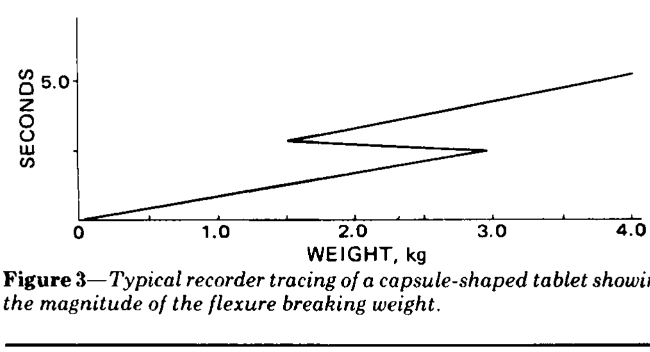

Figure 3—*Typical recorder tracing of a capsule-shaped tablet showing the magnitude of the flexure breaking weight.*

Table I—Typical Flexure Breaking Strength and Tablet Hardness Values (±*SD*)

Tablet Thickness[a], mm	Tablet Weight, mg	Flexure Breaking Strength ± *SD*, kg[b]	Tablet Hardness ± *SD*, kg[c]
6.299	588	1.25 ± 0.05	4.97 ± 0.08
6.325	620	1.97 ± 0.15	7.80 ± 0.21
6.325	650	2.79 ± 0.14	11.01 ± 0.66
6.350	680	3.99 ± 0.30	15.34 ± 0.78

[a] All tablets were 7.035 mm wide and 17.900 mm long. [b] Mean of six measurements. [c] Mean of 10 measurements.

After the breaking point, the backswing develops because the load is relieved abruptly and the cantilever beam rapidly rises until it contacts the plunger. Six replicate measurements were made, and the mean is reported.

Tablet hardness[4] measurements were made by the diametral compression of capsule-shaped tablets through their major axes. Ten replicate measurements were made, and the mean is reported. Typical tablet hardness and flexure breaking strength measurements and their relative standard deviations are listed in Table I. The tablet mechanical strength profile was obtained by a least-squares line plot of tablet hardness *versus* flexure breaking strength.

Materials used in this study were USP, NF, or pharmaceutical grade. Lactose tablets contained 89.5% direct-compression lactose, 10% cornstarch, and 0.5% magnesium stearate. Capsule-shaped tablets, 7.035 × 17.900 mm, were prepared with one punch of a rotary tablet press[5] at a rate of 24 tablets/min using one station to minimize tooling variations. Tablets of other sizes were prepared on a modified single-punch tablet press[6].

RESULTS AND DISCUSSION

The effects of four different tablet thicknesses on the mechanical strength profile of lactose capsule-shaped tablets are shown in Fig. 4. From this profile, it is apparent that, at the same tablet hardness, an increase in tablet thickness resulted in an increase in flexure breaking strength. At 10-kg tablet hardness, for example, tablets prepared at a thickness of 5.08 mm (line A) had a flexure breaking strength of 2.2 kg, whereas tablets prepared at a thickness of 7.62 mm (line D) had a breaking strength of 3.4 kg.

The effects of various tablet formulations on the mechanical strength profile of tablets prepared at a constant thickness were studied. Measurements on 10 formulations are shown in Fig. 5. The formulations were prepared by either direct compression or wet granulation, and an attempt was made to vary the type and concentrations of fillers, binders, disintegrants, and lubricants. The formulation and ingredients did not significantly alter the mechanical strength of the tablet. The data indicate

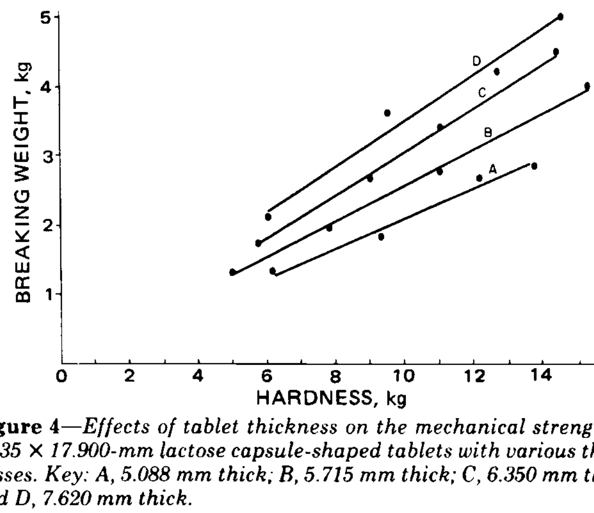

Figure 4—*Effects of tablet thickness on the mechanical strength of 7.035 × 17.900-mm lactose capsule-shaped tablets with various thicknesses. Key: A, 5.088 mm thick; B, 5.715 mm thick; C, 6.350 mm thick; and D, 7.620 mm thick.*

[1] Model 4K 868, Dayton Electric Manufacturing Co., Chicago, Ill.
[2] Model 4X 599, Dayton Electric Manufacturing Co., Chicago, Ill.
[3] Brown Electronik, Honeywell, Philadelphia, Pa.
[4] Heberlein model 2E/106, series 7203, Key Industries, Farmingdale, N.Y.
[5] Colton 216, Cherry-Burrell Corp., Park Ridge, Ill.
[6] Colton T-12, Cherry-Burrell Corp., Park Ridge, Ill.

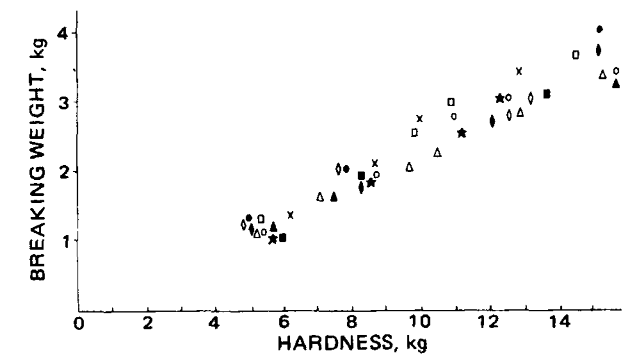

Figure 5—*Effects of 10 formulations on the mechanical strength of a 7.035 × 17.900 × 6.350-mm capsule-shaped tablet. Key: ●, direct-compression lactose (89.5%), cornstarch (10%), and magnesium stearate (0.5%); ×, unmilled dibasic calcium phosphate dihydrate (89.5%), cornstarch (10%), and magnesium stearate (0.5%); □, unmilled dibasic calcium phosphate dihydrate (44.75%), direct-compression lactose (44.75%), cornstarch (10%), and magnesium stearate (0.5%); ◇, direct-compression lactose (44.75%), microcrystalline cellulose (44.75%), cornstarch (10%), and magnesium stearate (0.5%); ○, unmilled dibasic calcium phosphate dihydrate (44.75%), microcrystalline cellulose (44.75%), cornstarch (10%), and magnesium stearate (0.5%); ★, unmilled dibasic calcium phosphate dihydrate (77%), microcrystalline cellulose (20%), and stearic acid (3%); ■, dibasic calcium phosphate granules prepared by wet granulation (89.5%), cornstarch (10%), and magnesium stearate (0.5%); ▲, calcium sulfate granules prepared by wet granulation (95%), sodium carboxymethyl starch (4%), and hydrogenated vegetable oil (1%); ♦, calcium sulfate granules prepared by wet granulation (89.5%), cornstarch (10%), and magnesium stearate (0.5%); and △, calcium sulfate granules prepared by wet granulation (64.5%), α-cellulose (25%), cornstarch (10%), and magnesium stearate (0.5%).*

that, for this specific tablet configuration, there is a linear relationship between the tablet crushing strength as determined by the tablet hardness measurement and the flexure breaking strength.

The mechanical strength of different size lactose tablets is shown in Fig. 6. At the lower tablet hardness values, the differences in breaking strengths between the different size tablets were small; at the higher tablet hardness values, the differences were significantly greater. The oval tablet (line D) had greater breaking strength than the capsule-shaped tablet. Since each breakage problem represents a unique set of conditions,

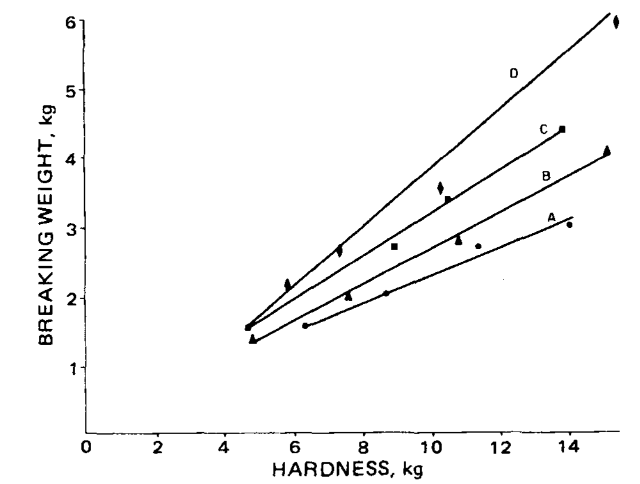

Figure 6—*Effects of different size tooling on the mechanical strength of lactose tablets. Key: A, tablet of 5.156 (width) × 16.612 (length) × 5.105 (thickness) mm; B, tablet of 7.035 (width) × 17.900 (length) × 6.350 (thickness) mm; C, tablet of 7.112 (width) × 22.987 (length) × 7.239 (thickness) mm; and D, oval-shaped tablet of 7.976 (width) × 19.177 (length) × 6.655 (thickness) mm.*

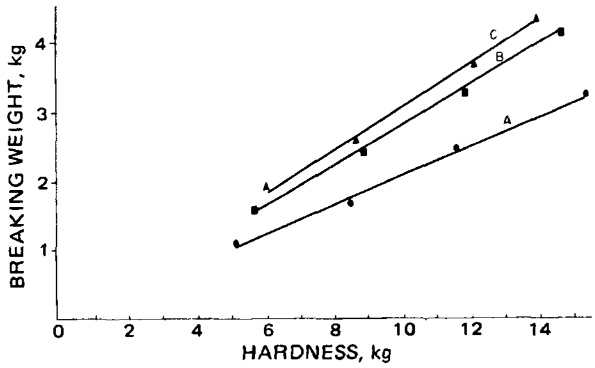

Figure 7—*Effects of tablet concavity on the mechanical strength of a 7.035-mm wide × 17.900-mm long × 6.350-mm thick lactose capsule-shaped tablet. Key: A, 1.143 mm deep; B, 1.8034 mm deep; and C, 2.235 mm deep.*

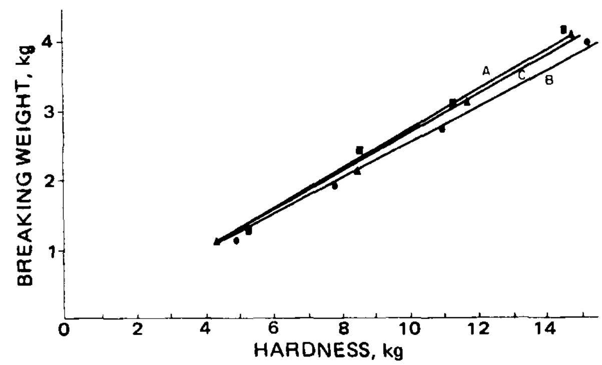

Figure 8—*Effects of the bisect bar dimensions on the mechanical strength of a 7.035-mm wide × 17.900-mm long × 6.350-mm thick lactose capsule-shaped tablet. Key: A, no bisect bar; B, 0.762-mm wide and 0.381-mm deep bisect bar; and C, 1.524-mm wide and 0.381-mm deep bisect bar.*

the size and configuration of the tablet must be evaluated in terms of the specific problem. Where the cross-sectional area of the tablet may be the critical parameter in coating tablets, the length may be the critical parameter in a bottle-filling operation.

The effect of the concavity of the tablet on the mechanical strength of lactose tablets was evaluated (Fig. 7). Tablets were prepared with shallow (1.143 mm), medium (1.803 mm), and deep (2.235 mm) cups in which the overall dimensions of the tablet were maintained at 7.035 × 17.900 × 6.350 mm. Tablets made with the shallow cup had significantly less breaking strength at the same tablet hardness than did either the medium or deep cup tablets. At 12-kg tablet hardness, the shallow cup had a breaking strength of only 2.4 kg compared to the standard cup value of 3.3 kg and the deep cup value of 3.6 kg.

The effect of the bisect on the mechanical strength of lactose tablets also was evaluated (Fig. 8). Using the same size tooling, tablets were prepared with no bisect, a 0.762-mm wide and 0.381-mm deep bisect, and a 1.524-mm wide and 0.381-mm deep bisect. Surprisingly, the bisect did not significantly alter the mechanical strength of these tablets.

REFERENCES

(1) C. J. Endicott, W. Lowenthal, and H. M. Gross, *J. Pharm. Sci.*, **50**, 343 (1961).

(2) S. T. David and L. L. Augsburger, *ibid.*, **63**, 933 (1974).

(3) H. Spengler and A. Kaelin, *Pharm. Acta Helv.*, **20**, 219, 239 (1945).

(4) K. Munzel and W. Kagi, *ibid.*, **32**, 305 (1957).

(5) G. Gold, R. N. Duvall, B. T. Palermo, and R. L. Hurtle, *J. Pharm. Sci.*, **60**, 922 (1971).