⟨1217⟩ TABLET BREAKING FORCE

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INTRODUCTION

There are a variety of presentations for tablets as delivery systems for pharmaceutical agents, such as rapidly disintegrating, slowly disintegrating, eroding, ▲and▲ (USP 1-May-2019) chewable. ▲The concepts of this chapter are also applicable to lozenges.▲ (USP 1-May-2019) Each of these presentations places a certain demand on the bonding, structure, and integrity of the compressed matrix. Tablets must be able to withstand the rigors of handling and transportation experienced in the manufacturing plant, in the drug distribution system, and in the field at the hands of the end users (patients/consumers). Manufacturing processes such as coating, packaging, and printing can involve considerable stresses, which the tablets must be able to withstand. ▲For these reasons, several tests are available to assess the strength of the compact as well as the response to contact with water and other liquids. These tests include *Disintegration* ⟨701⟩, *Dissolution* ⟨711⟩, *Tablet Friability* ⟨1216⟩, and *Tablet Breaking Force*, the subject of this chapter. The mechanical strength of tablets is of considerable importance and is routinely measured. Tablet strength serves both as a criterion by which to guide product development and as a quality control specification. An acceptable value for the strength of the compact must consider the intended use as well as the mechanism of release of the active ingredient(s) from the dosage form.▲ (USP 1-May-2019)

One commonly employed test of the ability of tablets to withstand mechanical stresses determines their resistance to chipping and surface abrasion by tumbling them in a rotating cylinder. The percentage weight loss after tumbling is referred to as the friability of the tablets. Standardized methods and equipment for testing friability have been provided in ⟨1216⟩. ▲In addition to the loss of active drug, chipping and abrasion can each have a significant impact upon the success of subsequent manufacturing operations such as coating and packaging, and impact the consumer's perceived elegance of the dosage form.▲ (USP 1-May-2019)

Another measure of the mechanical integrity of tablets is their "breaking force", which is the force required to ▲produce failure (i.e., breakage)▲ (USP 1-May-2019) in a specific plane. The tablets are generally placed between two platens, one of which moves to apply sufficient force to the tablet to cause fracture. For conventional, round (circular cross-section) tablets, loading occurs across their diameter (sometimes referred to as diametral loading), and fracture occurs in that plane. ▲As previously stated, friability and breaking force measure different aspects of the compact strength. Depending upon the formulation, manufacturing method, and intended use, the results from one of the tests may be a better indicator of the quality of the dosage form than the other. This determination is best made at the time the formulation and manufacturing process are defined.▲ (USP 1-May-2019)

The breaking force of tablets is commonly called "hardness" in the pharmaceutical literature; however, the use of this term is misleading. In material science, the term "hardness" refers to the resistance of a surface to penetration or indentation by a small probe. The term "crushing strength" is also frequently used to describe the resistance of tablets to the application of a compressive load. Although this term describes the true nature of the test more accurately than does "hardness", it implies that tablets are actually crushed during the test, which often is not the case ▲nor the intent of the breaking force determination.▲ (USP 1-May-2019) Moreover, the term "strength" in this application can be questioned, because in the physical sciences that term is often used to describe a stress (e.g., tensile strength). Thus, the term "breaking force" is preferred and will be used in the present discussion.

▲During manufacture of compressed tablets, decisions must be made regarding tablet weight, thickness, friability, and breaking strength targets. Control of tablet weights must be given priority because weight control directly correlates with dosing accuracy. Once the desired weight is achieved, decisions must be made concerning the relative importance of the thickness, loss on drying, friability, and breaking strength. While thickness may be viewed as a physical parameter that only influences the appearance of the tablet, it also determines the pore volume of the compact (i.e., solid fraction, see also *Tablet Compression Characterization* ⟨1062⟩). While thickness, friability, and breaking strength are related, the relationships are not clear or easily predicted. As illustrated in ⟨1062⟩, the thickness of the tablet at constant weight may not be varied without influencing the breaking strength. It is therefore important for the manufacturer to identify which parameter most closely correlates with desired product performance.▲ (USP 1-May-2019)

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TABLET BREAKING FORCE DETERMINATIONS

Early measuring devices were typically hand operated. For example, the Monsanto (or Stokes) hardness tester was based on compressing tablets between two jaws via a spring gauge and screw. In the Pfizer hardness tester, the vertically mounted tablet was squeezed in a device that resembled a pair of pliers. In the Strong-Cobb hardness tester, the breaking load was applied through the action of a small hydraulic pump that was first operated manually but was later motorized. Problems associated with these devices were related to operator variability in rates of loading and difficulties in proper setup and calibration. Modern testers employ mechanical drives, strain gauge-based load cells for force measurements, and electronic signal processing, and therefore are preferred. However, several important issues must be considered when using them for the analytical determination of breaking force; these are discussed below.

Platens

The platens should be parallel. Their faces should be polished smooth and precision-ground perpendicularly to the direction of movement. ▲In addition, the face of the platen should be perpendicular to the surface on which the tablet is placed to ensure the platen uniformly contacts the side of the tablet from top and bottom.▲ (USP 1-May-2019) Perpendicularity must be preserved during platen movement, and the mechanism should be free of any bending or torsion displacements as the load is applied. The contact faces must be larger than the area of contact with the tablet. ▲These considerations ensure that the applied load is operating over a consistent surface area of the dosage form.▲ (USP 1-May-2019)

Rate and Uniformity of Loading

Either the rate of platen movement or the rate at which the compressive force is applied (i.e., the loading rate) should be constant. Maintaining a constant loading rate avoids the rapid buildup of compressive loads, which may lead to uncontrolled crushing or shear failure and greater variability in the measured breaking force. However, constant loading rate measurements may be too slow for real time monitoring of tablet production.

The rate at which the compressive load is applied can significantly affect results, because time-dependent processes may be involved in tablet failure (*1*). How a tablet matrix responds to differences in the loading rate depends on the mechanism of failure.

▲Additional discussion of the consolidation mechanism of materials is provided in ⟨1062⟩. Since different formulations of tablets exhibit varying amounts of strain rate sensitivity, it is important to conduct the breaking force test in a controlled manner and recognize that instruments that operate with different platen movement or loading rates may not give comparable results for the same product.▲ (USP 1-May-2019)

The test must be run consistently with equipment that has been routinely calibrated. Changing from testing units of different designs or from different manufacturers will require comparison of data to ensure that the two units are subjecting the dosage form to similar stress in a similar manner. Currently available equipment provides a constant loading rate of 20 newtons (N) or less per second or a constant platen movement of 3.5 mm or less per second. Controlled and consistent breaking is an important test procedure attribute. To ensure comparability of results, testing must occur under identical conditions of loading rate or platen movement rate. Since there are certain advantages to each system of load application, both are found in practice. Because the particular testing situation and the type of tablet matrix being evaluated will pose different constraints, there is also no basis to declare an absolute preference for one system over the other. This general chapter proposes consideration of both approaches.

The different methods may lead to numerically different results for a particular tablet sample, requiring that the rate of load application or displacement must be specified along with the determined breaking force.

Dependence of Breaking Force on Tablet Geometry and Mass

Measurements of breaking force do not take into account the dimensions or shape of the tablet. ▲▲ (USP 1-May-2019) Tablet orientation and failure should occur in a manner consistent with ▲the orientation used and failure observed▲ (USP 1-May-2019) during the development of the dosage form. For direct comparisons (i.e., without any normalizations of the data), breaking force measurements should be performed on tablets having the same dimensions, ▲weights,▲ (USP 1-May-2019) geometry, and consistent orientation in test equipment.

Tablet Orientation

Tablet orientation in diametral compression of round tablets without any scoring is unequivocal. That is, the tablet is placed between the platens so that compression occurs across a diameter. However, tablets with a unique or complex shape may have no obvious orientation for breaking force determination.

▲Because the breaking force for unembossed, noncircular cross-section tablets will likely depend on the tablet's orientation in the tester, it is best to settle on a standard orientation, preferably one that provides a consistent failure plane and that is readily reproduced by operators, to ensure comparability of results.▲ (USP 1-May-2019) In general, tablets are tested either across the diameter or parallel to the longest axis. Scored tablets have two orientation possibilities. When they are oriented with their scores perpendicular to the platen faces, the likelihood that tensile failure will occur along the scored line increases. This provides information about the strength of the matrix at the weakest point in the structure. When scored tablets are oriented with their scores parallel to the platen faces, more general information about the strength of the matrix is derived.

Capsule-shaped tablets or scored tablets may best be broken in a three-point flexure test (*2*). A fitting, which is either installed on the platens or substituted for the platens, supports the tablet at its ends and permits the breaking load to be applied to the opposite face at the unsupported midpoint of the tablet. The fittings are often available from the same source that supplies the hardness tester.

▲Since the orientation of the dosage form critically impacts the result of the test, it must be clearly defined in the test procedure and produce a consistent failure plane in the dosage form for the results to be comparable. The plane of failure should always be noted. If, while maintaining orientation, the plane of failure differs for a batch or sub-batch of tablets relative to previous results and direct comparison of the numerical value is not possible, this observation in itself can be an important indicator of a compression problem.▲ (USP 1-May-2019)

Units, Resolution, and Calibration

Modern breaking force testers are usually calibrated in kiloponds or newtons. The relationship between these units of force (*3*) is 1 kilopond (kp) = 1 kilogram-force (kgf) = 9.80 newtons (N). The test results should be expressed in standard units of force which facilitate communication. Some breaking force testers also will provide a scale in Strong-Cobb units (SCU), a carryover from the days when Strong-Cobb hardness testers were in common usage. The conversion between SCU and N or kp must be viewed with caution, because the SCU is derived from a hydraulic device and is a pressure.

Generally, contemporary breaking force testers use modern electronic designs with digital readouts. Some units also have an integral printer or may be interfaced with a printer. Breaking forces should be readable to within 1 N.

Breaking force testers should be calibrated periodically. The force sensor as well as the mechanics of the apparatus needs to be considered. For the force sensor, the complete measuring range (or, at a minimum, the range used for measuring the test sample) should be calibrated to a precision of 1 N, using either the static or dynamic method. Static calibration generally employs traceable counterweights; at least three different points are checked to assess linearity. Dynamic calibration makes use of a traceable reference-load cell that is compressed between the platens. The functional calibration of a breaking force test apparatus should also confirm that the velocity and the constancy of velocity for load application or displacement are within prescribed tolerances throughout the range of platen movement.

Sample Size

▲Interpretation of the breaking force data must consider not only the mean value but the consistency of the test results for multiple tablets. The range of breaking force values may provide valuable information on the consistency of the tableting process. Excessive variability in values may reflect issues with die filling, weight control, or orientation of the dosage form in the test equipment. Additionally, the breaking strength result will reflect the orientation of the failure plane. For results to be compared, the tablets must fail in the same manner throughout the test. In cases where breaking force may be particularly critical, the average plus individual breaking force values should be reported.▲ (USP 1-May-2019)

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TENSILE STRENGTH

▲Tensile strength is an intensive materials property, which by definition does not depend on the size or shape of the material being measured. The measurement of tensile strength provides a more fundamental measure of the mechanical strength of the compacted material and takes into account the geometry of the tablet. The application of a vertical force (*F*), across the diameter of a disc-shaped tablet generates a horizontal stress (σ_X), which is tensile and constant along the centerline of the tablet. The value of this stress is given by the following equation, which applies to diametral compression of cylindrical tablets:

$$\sigma_X = 2F/\pi DH$$

σ_X = horizontal stress
F = breaking force
D = tablet diameter
H = tablet thickness

The derivation of this equation may be found in standard texts (*4*–*6*); it is based on elastic theory and the following assumptions:

1. The tablet is an isotropic body
2. Hooke's law is obeyed
3. The modulus of elasticity in compression and in tension is the same
4. Ideal point loading occurs

If the force is increased to the point of tablet fracture and if the tablet breaks into two roughly equal halves, the corresponding tensile stress at failure is historically reported as the "tensile strength". However, recent studies (*7*) show that the tensile strength calculated using this method can be half or less of the actual tensile strength as measured in a three-point bend test. The discrepancy occurs because tablet fracture initiates under the intense shear stresses (*8*) that are present near the point of contact between the tablet and the compression platen. The intense shear stresses allow cracks to form under Mode III fracture conditions (out-of-plane shearing), even though σ_X is still below the strength of the compact. Once fracture initiates, the cracks propagate along the center line by Mode I fracture (opening) and at tensile stresses well below the true tensile strength of a crack-free tablet. Despite these errors the diametrical strength is still useful in many applications (*9*). Moreover, the data will be normalized with respect to tablet dimensions, because both diameter and thickness are included in the equation.

The derivation has been extended to convex-faced tablets (*10*,*11*):

$$\sigma_X = (10F/\pi D^2) \times [(2.84H/D) - (0.126H/W) + (3.15W/D) + 0.01]^{-1}$$

σ_X = tensile stress
F = breaking force
D = tablet diameter
H = tablet thickness
W = central cylinder thickness (tablet wall height)

Other examples may be available in the literature (*12*).▲ (USP 1-May-2019)

Bending or flexure of tablets is another option for determining the tensile strength of tablets. Under ideal loading conditions, a breaking load applied to the unsupported midpoint of one face will result in the generation of pure tensile stress in the opposite face. If the tablets are right circular cylinders and are subjected to three-point flexure, the tensile strength may be estimated using the following equation (▲*13*▲ (USP 1-May-2019)):

$$\sigma_X = 3FL/2H^2D$$

σ_X = tensile stress
F = breaking force
L = distance between supports
H = tablet thickness
D = tablet diameter

▲The value of *L* should be chosen judiciously to minimize errors in this calculation (*9*).▲ (USP 1-May-2019) The assumptions are the same as those for calculating tensile strength from diametral compression. However, tensile strengths determined by flexure and diametral compression may not agree because of likely nonideal loading and the induction of shear failure during testing.

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