Measurement of Lower Punch Pulldown Force and Its Significance

FRANK W. GOODHART, GUSTAVO MAYORGA, and FRED C. NINGER

Abstract ☐ The technology for measuring forces occurring during the travel of lower punches over the lower pulldown cam of rotary tablet machines has been developed. This was accomplished by mounting strain gauges either internally or externally on the bolts holding these cams in place. Various formulations were monitored and it was found that binding forces were easily detected and were dependent on numerous factors. Among the factors were the extent of lubrication of both the formulation and machine, the cleanliness of the machine, and the length of running time.

Keyphrases ☐ Rotary tablet machines—lower punch frictional forces ☐ Punch, lower—pulldown force ☐ Diagram—lower punch pulldown force instrumentation ☐ Lubricant effect—frictional forces

The usefulness of compression and ejection measurements on rotary tablet machines has been well demonstrated by Knoechel (1, 2) and Wray (3). The relationship between the properties of compressed tablets, machine settings, and formulation has been found and described. Little work has been carried out to demonstrate the relationship between formulation and machine operation. It was thought that measurement of other forces within tablet machines might be of value in determining the smoothness of machine operation. The free movement of punches through rising and lowering cams is an important aspect of tablet-machine operation.

It has been shown that some of the more efficient and commonly used lubricants such as fatty acids and their salts are boundary types of lubricants (4). It is thought that the polar moiety of boundary-type lubricants is attached to the die-wall surface, thus providing a slipping interface between the compact and the die. Also of concern in tableting is the free movement of punches from the die immediately after compaction. Thus, another important facet of lubrication is the prevention of material from adhering to the punches and dies after compression. The antiadherence property of lubricants prevents sticking as well as adherence of material to the die wall. Freedom of punch movement after compression results in less tool and cam-track wearing and failure.

Frequently while making prolonged runs, upper and lower punches may begin to bind and stick either on the punch shank or at the punch tip and die-wall interface. Among conditions causing this sticking are inadequate removal of overflowing granulation, excessive binding properties of the material being tableted, and poor or faulty lubrication of the formulation or the machine. Strains resulting from the above conditions are often the cause of excessive punch and cam wear or tooling and machine failure.

It has been found that strain on cams may be measured by replacing one of the normal bolts with an identical bolt fitted with strain gauges. Tablet machines which employ segmented cams are generally held in place by two or three bolts. In the case of a lower-punch pulldown cam, the resulting force measurement reflects the friction of the lower punch-tip motion through the die and of the punch shank in its guide. Ejection forces, which have been described by other workers, measure a combination of forces, that of tablet ejection and lower-punch friction of both the tip and shank. Ejection forces are an indication of the effectiveness of formula lubrication while lower-punch pulldown force (LPPF) may indicate tooling and maintenance problems as well as formula characteristics. Therefore, the measurement of LPPF is another means of assessing frictional forces which sometimes cause generation of heat within tablet machines experienced with newer high-speed production equipment.

It is thought that similar measurements from upper cam tracks might be useful in measuring still other frictional forces during tableting.

EXPERIMENTAL

Instrumented Bolt for the Rotary Tablet Press A[1] Lower-Punch Pulldown Cam—The lower-punch pulldown cam on the tablet press[1] is held in place by two 1.27-cm. (0.5-in.) diameter bolts. Increased lower-punch binding should result in measurable strain on these bolts. Therefore, a bolt was instrumented with metal foil strain gauges as shown in Fig. 1. A hole was drilled partially into the center of the bolt. The thread near the head was machined off and a hole was drilled through the machined area to the center hole. Strain gauges, in a full Wheatstone-bridge configuration, were attached to the machined portion of the bolt and wire leads were threaded through the center hole. The instrumented bolt was tested by replacing one of the regular bolts used to hold the pulldown cam in place. Oscilloscopic readings were obtained using an oscilloscope[2] with a strain gauge amplifier. Photographs were taken using a self-developing camera.[3]

A hexagonal head screw[4] was installed and similar results to those found with the authors' installation were obtained. These internally strain-gauged bolts are available in a number of specifications and sizes.

Instrumented Bolt for the Rotary Tablet Press B[5] Lower-Punch Pulldown Cam—Three bolts are used to hold this cam in place;

---

[1] Stokes BB-2, 27-station press, Stokes Tabletting Equipment Department, Pennsalt Chemicals Corporation, Warminster, Pa.

[2] Tektronix 545A Oscilloscope and Type Q Strain Gage Amplifier, Tektronix Inc., Beaverton, Oregon.

[3] Polaroid, Model C-12.

[4] Strainsert Hex-Head Capscrew, Strainsert Co., Bryn Mawr, Pa.

[5] Manesty 45 Rotapress, Manesty Machines Ltd., Speke, Liverpool, England.

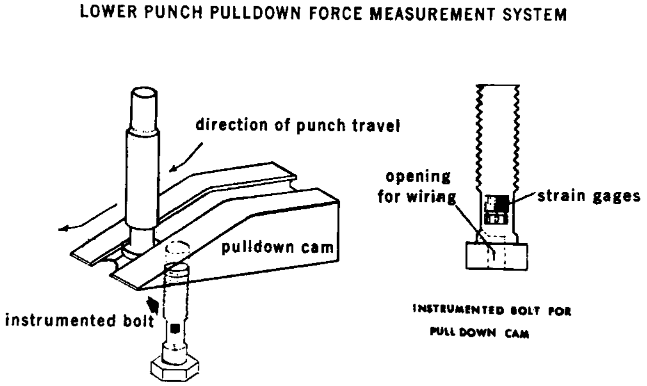

Figure 1—*Instrumental bolt measurement of lower-punch pulldown force (LPPF). See text for description.*

these bolts are 1.27 cm. (0.5 in.) in diameter (Whitworth threaded, 12 threads to the inch) and measure 5.08 cm. (2 in.) under the head. A bolt was instrumented with strain gauges in the same way as shown in Fig. 1 and tested for use in all three positions.

In order to fully test the usefulness of LPPF, the responses of the instrumented lower-punch pulldown cam were monitored on both machines during compression of a marketed antacid tablet formulation. A pilot scale-up of a different formulation of 500,000 tablets using rotary tablet press A was also monitored. This was a direct-compression formulation containing about 90 mg. of dicalcium phosphate per tablet and additional fillers to make a final tablet weight of 130 mg.

A series of experiments then was run in order to determine the relative effectiveness of three stearate-type of lubricants for mannitol granulations. The lubricants chosen were the stearates of magnesium, calcium, and zinc. This factorially designed experiment also included two lubricant levels, 0.5 and 1.5%, and two methods of lubricant addition. One method of lubricant addition, the dry method, involved blending the stearate with some fines, incorporation of the fines into the balance of the mix, and drum rolling for 5 min. In the wet method, the stearate was suspended in isopropanol and sprayed onto the moving bed of mannitol granulation. The mix was then vacuum-dried at 100°F. for 30 min. Tablets 1.59 cm. ($^{5}/_{8}$ in.) in diameter weighing 1.10 g. were compressed on the instrumented rotary tablet press A. LPPF and ejection forces were determined at 0, 5, and 10 min. The lubricants were examined microscopically in order to determine particle size and shape. An estimation of the particle size of zinc and calcium stearates was determined by use of the particle counter.[6]

RESULTS AND DISCUSSION

LPPF Using Rotary Tablet Press A—The machine was set up with 1.59-cm. ($^{5}/_{8}$-in.) flat-faced bevel-edged round punches and dies. Before tablets were made, the LPPF response was checked by running the machine at 20 r.p.m. without granulation. Upper and lower punch heads were lubricated with a special lubricant.[7] After several hours of running, no LPPF response was noted, thus indicating freedom from friction or inertial forces which might occur due to merely running the punches over the cams.

An upper punch was removed from one station and the hoppers were filled with granulation. Tablets weighing 1.30 g. were prepared using a lubricant level of 1.8%. The normal response of LPPF measurement under these conditions where binding does not occur is shown in Fig. 2. The upper photograph illustrates LPPF at low pressure and the lower at higher pressure. The flat tracing on the two photographs shows the station containing a lower punch and die but no upper punch (no tablet was produced at this station). It was found that smooth uniform waves of this nature are obtained when no binding is evident. The increased amplitude for tablets compressed at higher pressure results in a higher LPPF. Average μstrain readings of 6 and 10 were obtained for the lower and higher compression forces, respectively.

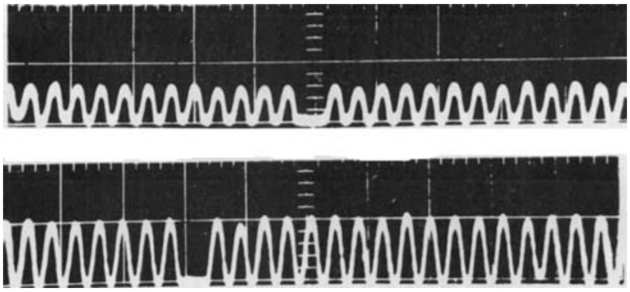

Figure 2—*Normal response of LPPF when no binding occurs. Upper tracing, 6 μstrain, obtained at low pressure. Lower tracing, 10 μstrain, obtained at high pressure. Flat part of tracing indicates station at which no tablets were produced.*

Figure 3 shows the relationship of ejection force obtained during these trials for both high- and low-pressure settings. The higher ejection forces for the higher pressure settings have been reported previously (2). Thus, it can be expected both higher LPPF and ejection force will occur at higher compressive forces. This fact may be attributed to higher particle fracture and exposure of new surfaces under high pressure. New surfaces are not lubricated, hence greater material-die wall adhesion occurs. Also, it has been shown that the die wall itself is under considerable strain during tablet compression (5). The die, in absorbing a significant amount of applied force, is prone to pick up the material in a manner similar to picking and sticking of punches. The picking up of material by the die or punch-tip surfaces results in higher cam strains as shown by LPPF and ejection-force measurement.

Figure 4 demonstrates the LPPF pattern of an identical granulation, but containing no lubricant and 0.1% lubricant. The granulation containing no lubricant binds immediately, and the resultant picture was taken after only one revolution of the tablet machine. The granulation containing 0.1% lubricant began to bind after several revolutions.

A pilot run of 500,000 tablets was monitored periodically for compression force, ejection force, and LPPF. The punches and dies were 0.63-cm. (0.25-in.) square and were therefore keyed. Tablets weighing 130 mg. were compressed at an average force of 1,250 lb. The standard deviation for compressive force was 83.5 lb. at the start of the run and 66.2 lb. at the end. Compressive force at the beginning of the run and at the end was not significantly different. Figure 5 illustrates the LPPF at the beginning and at the end of the

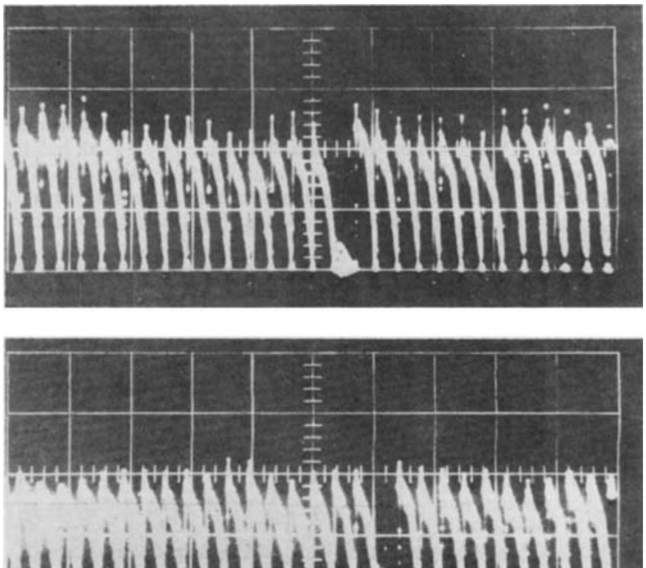

Figure 3—*Ejection force measurements. Upper tracing: high pressure. Lower tracing: low pressure.*

---

[6] Coulter Counter, Industrial Model B, Coulter Electronics, Chicago, Illinois.

[7] Ore-Lube, Ore-Lube Corp., College Point, N. Y.

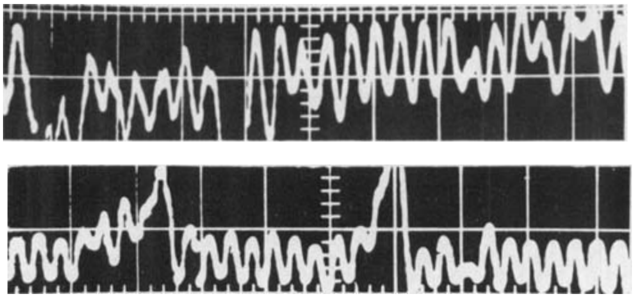

Figure 4—*LPPF pattern for granulation containing no lubricant (upper tracing) and 0.1% lubricant (lower tracing).*

run. The responses at the beginning are smooth and uniform. However, it was noted that near the end of the run four distinctive higher peaks occurred indicating significant lower punch hang-up at these positions. It was noted that in this case the ejection forces for these four stations were not higher than those of the nonbinding positions. No correlation between tool dimensions and binding was attempted. It is of importance to determine whether this binding effect was random or related to individual tool dimensions.

LPPF Using Rotary Tablet Press B—The instrumented bolt for the rotary tablet press B was found to respond best on the position closest to the ejection cam. Smooth signals were obtained at the beginning of a run of 1.59-cm. ($^5/_8$-in.) FFBE tablets. After running for 40 hr., it was found that LPPF increased to about 10 times the original value and that the responses became less uniform. One punch was identified as binding worse than the others as noted by an isolated higher peak. It was observed that the machine was warm and that some granulation was spilling over into areas traveled by the lower punch. The utility of LPPF measurement immediately became evident since further running of the machine in this condition could have resulted in breakdown of some components.

Lubrication Experiments on Mannitol Granulation—It was theorized that the wet process of lubricant distribution might be more efficient because of better distribution and coverage. Little attention has been given to lubricant addition methods or to the relationship of particle size of the lubricant to its efficiency. It has been reported that length of mixing time influences ejection force (3).

Average ejection forces for the various systems studied are given in Fig. 6 and LPPF results are shown in Fig. 7. In Fig. 6, it can be seen that the average ejection force for the wet process is generally higher than that for the dry process. An exception to this was the comparatively identical ejection forces for the dry- and wet-process calcium stearate at the 1.5% level. The overall ejection force variability of the wet *versus* the dry process is very significant. This would indicate better lubricant homogeneity in the dry process as compared with the wet. In general, magnesium and calcium stearates show decided advantages over zinc stearate for lubrication of mannitol granulation.

Figure 7 illustrates the number of punches out of six monitored that give a LPPF response. "Response" is defined by a peak height of at least 2 μstrain. Those conditions which showed a lack of LPPF response over the 10-min. testing period were: (*a*) calcium stearate, 1.5% concentration, both wet and dry process; and (*b*) magnesium stearate, 1.5% concentration, dry process only. These results are in agreement with the three lowest sets of ejection responses seen in Fig. 6. Definite differences can be seen between concentration of lubricant and length of running time. In formulating new products both ejection force and LPPF can be minimized by running experiments of this type.

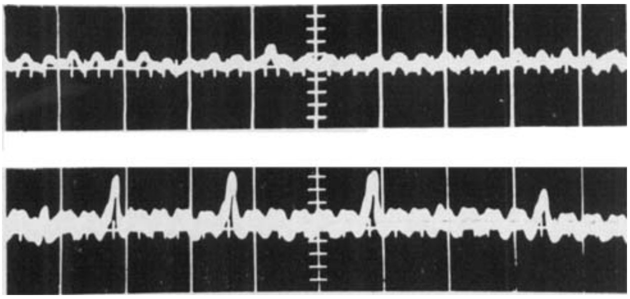

Figure 5—*LPPF pattern at the beginning (upper tracing) and end (lower tracing), of a pilot run of 500,000 tablets. Average compression force was 1,250 lb. and the tooling was 0.63 cm. (¼-in.) flat-faced bevel-edged square.*

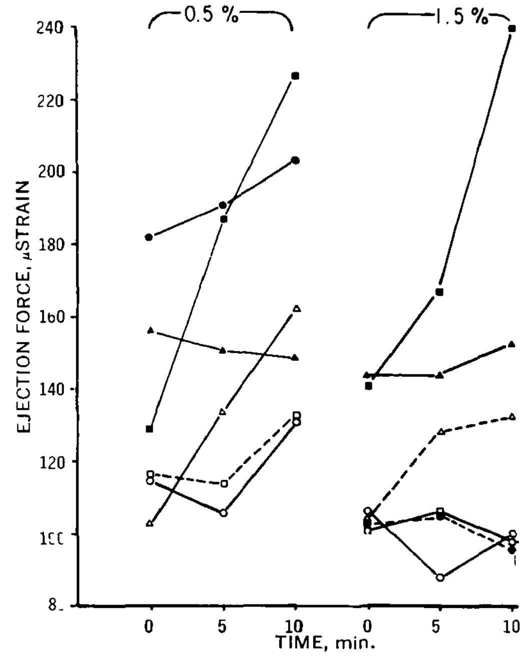

Figure 6—*Average ejection force of mannitol granulation lubricated with various stearates. Key:* ■, *magnesium stearate, wet process;* □, *magnesium stearate, dry process;* ●, *calcium stearate, wet process;* ○, *calcium stearate, dry process;* ▲, *zinc stearate, wet process;* △, *zinc stearate, dry process.*

At the end of each run, the tooling was removed and cleaned. It was always found that those stations showing LPPF response had either the punches or the die (or sometimes both) coated with adhering granulation. Those stations having no adhering granulation did not give any LPPF response.

All three stearates tested are substantially insoluble in isopropanol. Particle-size analysis by particle counter indicated that

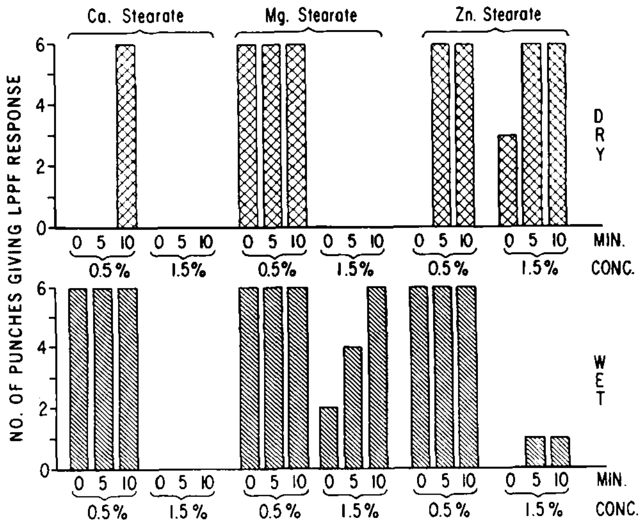

Figure 7—*LPPF response of mannitol granulation lubricated with various stearates. Six punches were monitored and the number giving at least a 2-μstrain response was noted. Note results for both wet and dry process as well as the two concentrations of lubricant employed.*

zinc stearate had an average particle size of less than 9 μ and that calcium stearate average particle size was substantially less than this. Magnesium stearate ranged from 10–50 μ by microscopic examination and showed many long, feathery type of particles. Whether these differences in particle size are of significance in the lubrication efficiency has not yet been determined. The other factor in question with regard to the wet process is the degree of dispersibility of the stearates in isopropanol.

CONCLUSIONS

The measurement of lower-punch pulldown force (LPPF) has been demonstrated on the two rotary tablet presses. The LPPF measurement is a qualitative or semiquantitative means of detecting changes in frictional forces involving lower-punch movement. It has utility in assessing frictional aspects of tooling, formula lubrication, and aids in prevention of machine breakdown or excessive tool wear. Similar research on upper cam-track measurement is now in progress.

REFERENCES

(1) E. L. Knoechel, C. C. Sperry, H. E. Ross, and C. J. Lintner, *J. Pharm. Sci.*, **56,** 109(1967).

(2) E. L. Knoechel, C. C. Sperry, and C. J. Lintner, *ibid.*, **56,** 116(1967).

(3) P. E. Wray, J. G. Vincent, F. W. Moller, and G. J. Jackson, paper presented to the Industrial Pharmacy Section, APhA Academy of Pharmaceutical Sciences, Dallas meeting, April 1966.

(4) W. O. Strickland, Jr., *Drug Cosmetic Ind.*, **85,** 319(1959).

(5) J. J. Windheuser, J. Misra, S. P. Eriksen, and T. Higuchi, *J. Pharm. Sci.*, **52,** 767(1963).

ACKNOWLEDGMENTS AND ADDRESSES

Received May 22, 1968, from the *Pharmaceutical Research and Development Laboratories, Warner-Lambert Research Institute, Morris Plains, NJ 07950*

Accepted for publication October 17, 1968.

NOTES

Determination of Hexachlorophene in Whole Blood

PETER J. PORCARO, PETER SHUBIAK, and MILTON MANOWITZ

Abstract □ An efficient method for the quantitative recovery of hexachlorophene from whole blood is described. It can be applied to both human and animal blood, and utilizes an extraction procedure followed by electron-capture detection with gas chromatography. Problems encountered in the reproducible isolation of hexachlorophene are discussed. Recoveries are in the 90–100% region with detectable levels down to 0.03 p.p.m. No blank interferences are observed.

Keyphrases □ Hexachlorophene—analysis in blood □ Extraction procedure—hexachlorophene in blood □ GLC electron-capture—analysis

A method for the quantitative analysis of hexachlorophene in animal tissues including blood has previously been described (1). The extraction and cleanup techniques were time consuming, and the lower limit of sensitivity was approximately 20 mcg. A recent publication demonstrated that hexachlorophene could accurately be determined at subnanogram levels using electron-capture gas chromatography (2). An efficient procedure for extracting hexachlorophene from blood and an improved analysis of the extract by electron-capture gas chromatography is described in this report.

EXPERIMENTAL

Apparatus—A gas chromatograph[1] equipped with a helium-discharge electron-capture detector was used in this work. A mixer[2] using 40-ml. round-bottom centrifuge tubes proved suitable for the extractions.

Reagents—Anhydrous ethyl ether, reagent grade hexane, and Tri-Sil concentrate[3] are used.

Procedure—*Calibration Curve*—One gram of hexachlorophene was dissolved in 100 ml. of ethanol. Ten microliters of this solution was diluted to 10 ml., and this dilution was used to prepare the calibration curve. Aliquots of 20, 40, 60, 80, and 100 μl. were evaporated to dryness and 10 μl. Tri-Sil concentrate was then added to the residue, followed by 1 ml. of hexane. One-microliter volumes were injected into the chromatograph. A calibration curve was prepared by plotting concentration *versus* recorder response (I_B background current) (2). The assay was thus calibrated from 0.2–1.0 ng./μl.

Recovery from Blood—Known amounts of hexachlorophene in alcoholic solution, as in the calibration procedure, were added to 3 ml. of whole blood contained in 40-ml. round-bottom centrifuge tubes. Seven milliliters of distilled water was then added to thin the blood and facilitate the thorough incorporation of hexachlorophene into the blood. This was followed by five successive 10-ml. extractions with ethyl ether, using the mixer. The blood-ether layers

---

[1] Beckman GC-5, Beckman Instruments, Inc., Fullerton, Calif.

[2] Vortex Genie, Scientific Industries, Inc., Springfield, Mass.

[3] Pierce Chemical Co., Rockford, Ill.