Development of IPT Standards for Tableting Tools

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Abstract □ A program of industry-wide standardization of punch and die specifications has been proposed by the Industrial Pharmaceutical Technology Section of the Academy of Pharmaceutical Sciences. The standards presented can be employed as guides for purchasing, incoming inspection, and manufacturing use of these tools. An outline for an incoming inspection program for tableting tools is also described.

Keyphrases □ Industrial Pharmaceutical Technology Committee report—punches and dyes □ Tableting tools—quality control standards □ Diagrams—punch and dye standardization □ Punches and dyes—standard specifications

The necessity for a comprehensive quality control program for tableting tools and the establishment of tooling standards and inspection facilities have been apparent for many years to a large segment of the pharmaceutical industry. Its importance was clearly recognized by the Industrial Pharmaceutical Technology (IPT) Section of the Academy of Pharmaceutical Sciences when in May, 1966, the Committee on Specifications was established. The defined purpose of this Committee was the development of standards for equipment, processes, and test procedures in areas of mutual concern and interest to pharmaceutical research, development, and production technologists.

As its initial endeavor, the Committee has established a collaborative study with suppliers of tableting tools to evolve industry-wide standards for punch and die specifications. These standards can then be used as guides for purchasing, incoming inspection, and manufacturing use of these tools.

It is recognized that there will always be a need for special tolerances and clearances due to a particular granulation or machine idiosyncracy and for special tablet shapes and designs for product identification. However, it was the opinion of the Committee that the bulk of the round-shaped tooling used in the industry could be standardized to the point of complete interchangeability regardless of supplier. Among other advantages, this would permit off-the-shelf deliveries which could materially reduce the user's inventory load.

As noted by Gaskell (1), "The supplier who manufactures punches and dies in large quantities would like to standardize on clearances for a great majority of his orders. He can take advantage of standardization to carry stock of finished punches and dies to enable him to give quick delivery and help keep down manufacturing costs by producing large quantities. The very fact that a manufacturer stocks finished punches and dies is, in itself, a guarantee that all punches and dies of a given tip diameter are interchangeable."

Following extensive intra-company correspondence and discussions with suppliers, a series of face-to-face meetings were held with the major suppliers of tableting

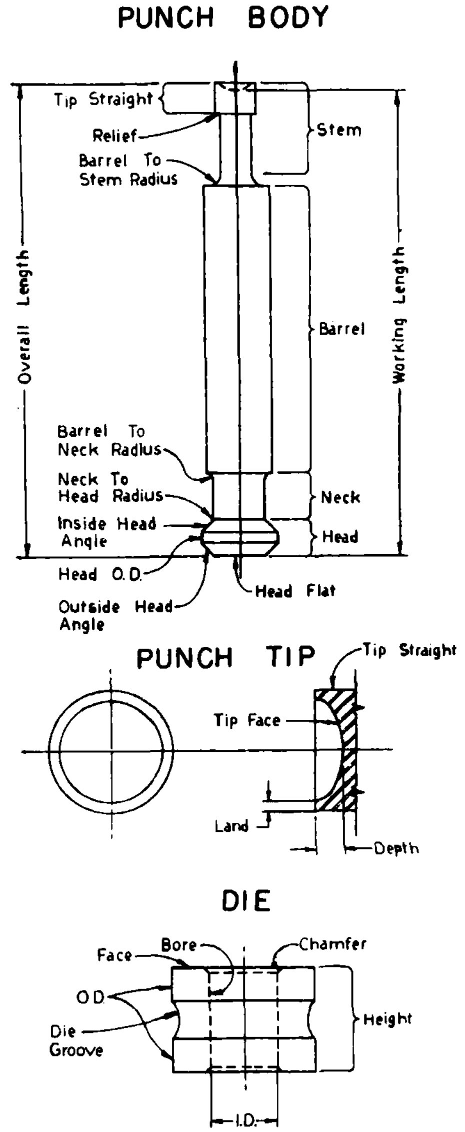

Figure 1—*Standard punch and die nomenclature.*

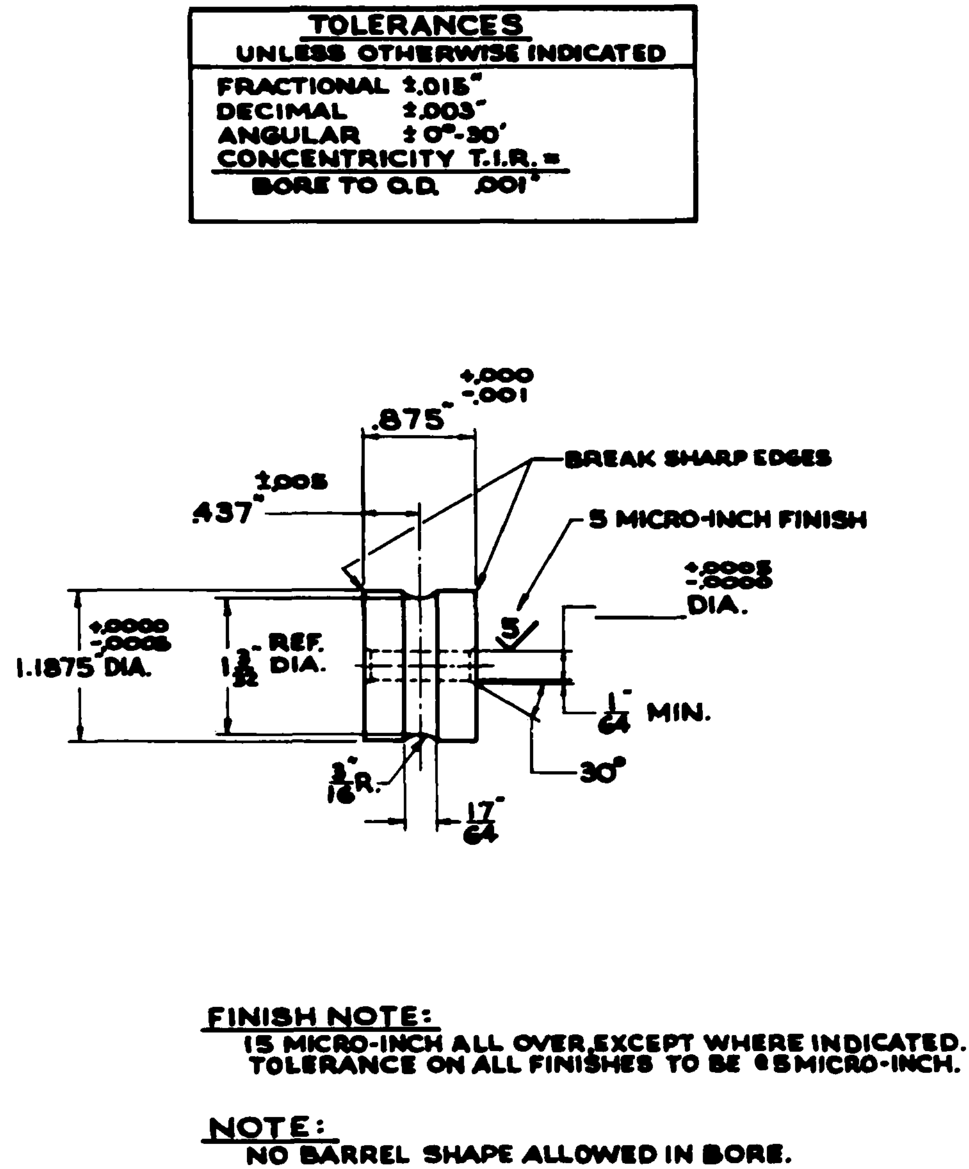

Figure 2—*Standard upper punch, 2.54 cm. (1 in.) diameter.*

tools to iron out differences in specifications and tolerances and arrive at a mutually satisfactory set of working drawings for the most commonly employed tools. In order to establish appropriate communications with the suppliers, a nomenclature drawing was developed which defined all working dimensions and eliminated ambiguity in data correlation. This drawing is presented as Fig. 1.

NEED FOR STANDARDS

Problems caused by tooling made with improper specifications can take many forms. Punches and dies that are dimensionally incorrect can cause numerous tableting problems that may affect tablet quality and production rates, and may sometimes result in serious damage to the tablet press and tools. Some of these difficulties are listed below.

1. Insufficient clearances between punch and die can cause tablet capping or laminating.
2. Insufficient clearances can result in severe binding of the punches in the punch socket or die bores that could ruin punches and cams very quickly.
3. A slightly barrel-shaped die bore can cause tablet capping and laminating as well as damage to the punch heads and ejection cams due to higher than normal ejectional loads.
4. Excessive variation in the overall length of punches can affect individual tablet weights and densities.
5. Adjustment of tableting pressures to suit a shorter punch in a set can result in overload damage.

STANDARDIZATION PROGRAM

Because of reasons such as noted above, the Committee on Specifications proceeded with the development of a series of standard tableting tool drawings. Representative examples of these drawings are presented as Figs. 2 and 3. It is intended that these drawings be employed as working prints in the routine ordering, manufacture, and control of tools.

The establishment of the tool specifications and tolerances for punches and dies as noted above was the first step. In order to ascertain the needs of the various pharmaceutical manufacturers, a questionnaire was sent to over 40 companies. A summary of each participating company's activities related to tableting tool specifications and tolerances, inspection and use was obtained as a base line for the standardization program. It can now be stated that the specifications and toler-

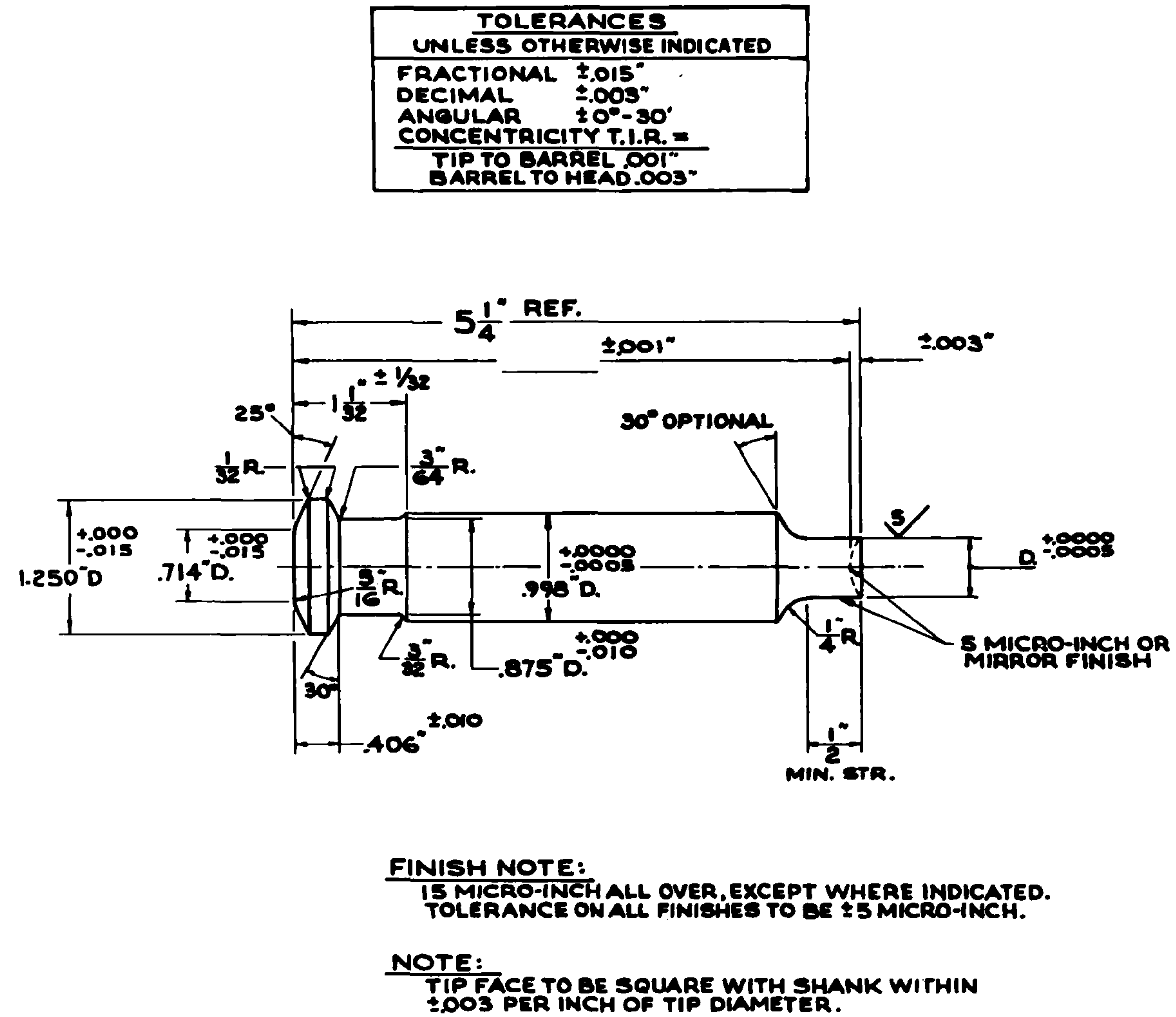

Figure 3—*Standard die, 3.05 cm. (1.1875 in.) diameter.*

ances above are acceptable to both the pharmaceutical industry and the tool suppliers. They are based on the requirements of the machines used, the products manufactured and the working tolerances of the supplier industry.

The most commonly used tools were standardized in this initial endeavor and include the following: 1.91 cm. (0.75 in.) diameter upper and lower punches; 2.54 cm. (1 in.) diameter upper and lower punches; 2.29 cm. (0.945 in.) diameter standard dies; 3.05 cm. (1.1875 in.) diameter standard dies; and 3.81 cm. (1.500 in.) diameter standard dies. In order to facilitate the use of the drawings, an interchangeability table for rotary tablet presses has also been prepared and is presented in Table I. It was felt that additional standardization of punch tips would be of further value in completing the preparation of IPT standard tools. As a result, representative proposed standard tip configurations for standard concave and deep concave tooling have been prepared.

It was the opinion of the Committee that on cupped punches, there should be a smooth, arithmetical progression of cup depth and the land on the punch face. The actual radius that allows for this land at a specified depth was designated. It is hoped eventually that flat-faced beveled-edge punches and bisections will be treated in a similar manner under the Committee aegis.

Although the drawings shown permit the preparation of tools with standard specifications and tolerances, a recommendation for a standard steel has not, as yet, been made by the Committee. Insufficient information is available at the present time to make such a judgment. The Committee is considering the establishment of appropriate techniques for steel comparisons. If a collective approach to this problem is made, it should be possible to preselect the best of the currently available steels for a particular compacting condition. Tool life records, to be described below, will be critically important in this area. Information on any given drug product being compressed will help to form a value judgment of appropriate steels and should include the following: (*a*) Type of steel, (*b*) hardness of steel, (*c*) cup shape—embossed or not embossed, (*d*) compaction pressure used, (*e*) number of tablets run per station before any failure in the set occurred, (*f*) nature of this failure, and (*g*) abrasivity and corrosivity of the material to be compressed.

TABLETING TOOL CONTROL AND INSPECTION PROGRAM

Together with the above standard drawings, and almost equally important, a punch and die control and preventive maintenance program are being drawn up. This program will define an incoming tool inspection setup without which the finest standards are of no value.

Tools manufactured in accordance with standard dimensions and tolerances should be retained in individual complete sets as received from the supplier for better accountability. Control of the system will eventually provide invaluable information on tool life for prorating tool costs or modifying tool specifications for increased life or optimum performance.

INCOMING INSPECTION

In order to control the quality of the punches, it is necessary to control all related matters as well. It is the recom-

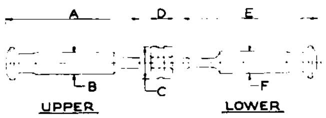

Table I—Interchangeability Table for Rotary Tablet Presses

Colton	Stokes	Manesty	A	B	C	D	E	F	Remarks
212,[a] 213,[a] 216–16, 204	B2–16	Betapress 16	5.250	0.748	1.1875[a]	0.875	3.562	0.745	SLP[b]
		Betapress 23	5.250	0.748	0.945	0.875	3.562	0.745	SLP[b]
227, 232, 233, 242–33,[a] 243–33[a] 247–33	BB2–27, 538, 540–35, 566–27	B3A–16, B3B–16, BB3B–27 Betapress 16 Layerpress 39 Rotapress 45	5.250	0.748	1.1875[a]	0.875	5.250	0.745	
233, 241, 242–41, 243–41,244 247–41	BB2–33 & 37, 540–41, 551–51	B3B–23, BB3B–33 & 39 Betapress 23 Layerpress 47 Rotapress 55	5.250	0.748	0.945	0.875	5.250	0.745	
240–16	D3–16	D3A–16, D3B–16, D3RY–16 Drycota 350 & 500–16 Bicota 500–16 Deltapress 16 Rotapress 29 & 37	5.250	0.998	1.500	0.9375	5.250	0.995	
250–12	DS–3		5.250	1.248	2.000	1.250	5.250	1.245	
260–25	DD2–23		6.812	1.248	2.125	1.500	8.812	1.245	
260–31[a]			6.812	1.248	1.750[a]	1.000	8.812	1.245	
260–33[a]			6.812	0.998	1.500[a]	1.500	8.812	0.995	
260–43[a]			6.812	0.998	1.1875[a]	1.500	8.812	0.995	
	DD2–31		6.812	0.998	1.500	1.250	8.812	0.995	
		D3RY–23 Drycota 900–23 Bicota 900–23	5.250	0.998	1.1875	0.9375	5.250	0.995	
	533–45		6.062	1.123	1.3125	1.500	6.250	1.120	
		Deltapress 23	5.250	0.998	1.1875	0.875	5.250	0.995	
	B2–22		5.250	0.748	0.945	0.875	3.562	0.745	SLP[b]

[a] Die o.d. differs (Colton M/C only). [b] Short lower punch.

mendation of the Committee that drawings such as those prepared be sent to the tooling manufacturers together with requests for purchase of tools. When the tools arrive from the manufacturer, a selective inspection program of incoming punches and dies is recommended. Engineers and mechanics utilizing appropriate instruments can employ techniques described below for checking all critical dimensions (2).

The measuring tools include an indicating micrometer, gauge blocks, an indicating bore gauge, and a master form gauge capable of measuring to an accuracy range of ± .0254 mm (0.001 in.). Other inspection devices include a depth-measuring stereo microscope to measure punch-face characteristics as well as a steel-hardness tester for Rockwell measurements. If the complete set of punches or dies meets all the standards, it is then accepted and can be etched with a control number for identification. If they fail to meet specifications, the faulty tools should be rejected and returned to the manufacturer for reprocessing or replacement. A typical incoming inspection report is presented here as Fig. 4.

Punch and Die Inspection Report

Control No.: Purchase Order No.:

Upper Punch Dwg. No.:	Lower Punch Dwg. No.:	Die Dwg. No.:
Monogram	Monogram	I.D.
Tip dia.	Tip dia.	O.D.
Shank dia.	Shank dia.	Height
Head	Head	Eccentricity
Lgth. (conc.-head)	Lgth. (conc.-head)	Hardness
Lgth. (tip-head)	Lgth. (tip-head)	Chrome
Depth of conc.	Depth of conc.	Control no.
Eccentricity	Eccentricity	
Hardness	Hardness	
Chrome	Chrome	
Control no.	Control no.	

Remarks ______________________________

____

Date Received ______ Started ______ Completed ______ Inspected by

Figure 4—*Punch and die inspection report.*

An accurate knowledge of punch and die dimensions can be attained by setting up a punch and die storage area where all tableting tools can be inspected before use and periodically during use to assume that certain critical dimensions are retained. The check for compliance with specifications might include the following steps:

1. Standard micrometer calipers can be used for checking all outside diameters.
2. The overall punch length, concavity depths, and die heights can be quickly and easily checked with an inexpensive dial indicator mounted on a comparator stand.
3. With V-blocks and a holder, the dial-indicating micrometer can also be used for checking concentricity, squareness, and the contour of most average-sized die bores.
4. The overall punch lengths can also be checked with a standard micrometer barrel mounted on a suitable stand. This arrangement would require an assortment of various-sized ball bearings for gauging from the bottom of the concavity of the punch face.
5. The die-bore size can be checked with an expanding-type small-hole gauge. A more expensive air-flow gauge[1] would provide maximum accuracy and efficiency and would be useful in cases of large volume inspection.
6. A lower power erect-image microscope will be helpful in checking embossed punch faces and detecting fine hairline cracks. However, a more expensive binocular microscope would speed up visual inspection of finishes and embossing.
7. Various types of dial indicators can be set up with V-blocks on a surface plate for checking several dimensions at once. With large-volume inspection such an arrangement would considerably reduce the inspection time.

[1] Compact Precisionaire instrument, Sheffield Corp., Cleveland, Ohio.

8. For maximum accuracy, a profilometer could be used for inspecting all finishes to the nearest micro-inch.

9. An optical comparator would also be of value since it would permit the precision inspection of all intricately shaped or hobbed punch faces.

10. A Rockwell or similar hardness tester can be used for checking punch and die hardnesses.

RECORDING TOOL LIFE HISTORY

The establishment of specifications and subsequent quality control of the tools should promote the purchasing of tools with a uniform high quality, But, at the present level of knowledge in this area, one cannot be certain that the quality of tooling now used, however uniform, is the most satisfactory for uninterrupted production. So that such information might be obtained, a system for recording the life of the various tools is suggested which will enable one to know the total number of tablets compressed with any given set of tools (3). In addition, information on the products prepared, machines the tools were used with, and the reasons that the tools were eventually discarded is recorded. The careful evaluation of the facts obtained from these records will be useful in purchasing tools with optimum wear characteristics and will result in a better finished product at reduced cost.

SUMMARY

An appropriate set of dimensional specifications and tolerances as well as an incoming inspection program for tableting tools can be considered the keys to an efficient tableting operation. The efforts of the IPT Committee on Specifications in establishing standard punch and die dimensions to suit the majority of pharmaceutical applications is a valuable contribution toward this goal. It is hoped that wide-spread acceptance of these standards will be forthcoming from the pharmaceutical industry since their advantages are self-evident.

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Automated Determination of Ascorbic Acid in Multivitamin Preparations

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Abstract ☐ A specific colorimetric method for the determination of ascorbic acid in multivitamin preparations has been automated. The method is based on the coupling reaction of diazotized 4-methoxy-2-nitroaniline with ascorbic acid. A bathochromic shift in the chromophore is effected by the addition of alkali. The stable blue color developed is measured at 570 mμ. Application of the automated procedure for the determination of ascorbic acid in multivitamin and mineral preparations is described.

Keyphrases ☐ Ascorbic acid in multivitamin products—automated analysis ☐ Automated procedure—ascorbic acid analysis ☐ Schematic diagram—ascorbic acid automated analysis ☐ Colorimetric analysis—spectrophotometer

Automated modified reactions of ascorbic acid with 2,6-dichlorophenol-indophenol, measured in aqueous media or an organic solvent extract, have been reviewed by Khoury (1). The indophenol reaction lacks specificity and is subject to interferences from reducing substances. Pelletier and Morrison (2) removed interfering ferrous and stannous ions with preliminary oxidation followed by chelation with EDTA before reaction of ascorbic acid with 2,6-dichlorophenol-indophenol. Robinson and Stotz (3) eliminated the interference of the reducing substances by peroxide treatment and the sulfhydryl groups by formaldehyde condensation.

Roe and Kuether (4) oxidized ascorbic acid to dehydroascorbic acid and then condensed it with 2,4-dinitrophenylhydrazine. This reaction is not subject to interference from reducing substances, however, difficulties arise from oxidizing substances such as ferric ions and hydrogen peroxide. Thiourea was used to maintain a reducing environment.

The high specificity of the coupling reaction of diazotized 4-methoxy-2-nitroaniline with ascorbic acid, reported by Schmall *et al.* (5, 6), along with the simplicity of the colorimetry, made it particularly suitable for an automated procedure. Dehydroascorbic acid, all other vitamins, and reducing agents such as ferrous and stannous ions do not interfere when present in quantities normally encountered in pharmaceutical preparations. The excipients commonly encountered in multivitamin preparations not reported by Schmall *et al.* (5) such as mannitol, talc, stearic acid, and magnesium