A Programmed Automated Film-Coating Process

By LEON LACHMAN and JACK COOPER

The design and operation of a programmed automated tablet coating process[1] is described. The electronics of the programmer, the baffle design for the coating pan, and the spray equipment used are illustrated and discussed. The advantages and superiority of this process as compared with the customary manual coating techniques are presented.

DURING THE PAST several years there has been a continuous and rapid movement away from the time-honored sugar coating to film coating of tablets. Considerable research time has been devoted to the development and testing of water- and gastric-soluble (1–11) and enteric (12–19) film-coating formulations. However, during this same period of time, little has been done to improve the process of applying the film coating onto tablets to insure reproducibility of the physical appearance and disintegrating characteristics of coating from batch to batch of tablets. The few attempts in this direction consisted of using a pneumatic gun to spray the tablets manually with coating solution (20) or the use of fluidized bed–coating techniques (21–23). Both methods, while being improvements over the customary manual technique of coating, still left something to be desired.

Most of the film coats presently on the market are of the water-soluble type which, in part, is the cause of the lack of progress in improving the coating process. Uniformity of water-soluble coating from batch to batch of tablets is of less importance than for enteric film coats. Satisfactory physical appearance and sufficient coverage to mask an unpleasant odor or taste are the major criteria for water- or gastric-soluble film coats. However, it is essential that the enteric film coating be uniform from batch to batch of tablets since the coated tablets must meet certain required disintegrating properties (U.S.P. XVI, p. 935) which are critically affected by film uniformity.

It is generally recognized that the human element is responsible for the variations that take place in batches of film-coated tablets. The thinness of the film coats, generally 1–10% of the uncoated tablet weight, cannot tolerate human variations or errors from batch to batch of tablets, which are known to occur in the customary sugar-coating technique. However, since sugar coats are usually equal to the weight of the uncoated tablet, the variations and errors due to the human factor become less meaningful.

In film coating, and more particularly in enteric film coating, it is imperative to remove the "art" from the coating process and place it on a more exact basis. This could most readily be accomplished by eliminating or minimizing the human factor in the coating process.

This report presents a detailed description of a programmed automated process for film coating of tablets recently developed in our laboratories which completely removes the human factor from the coating operation. In addition, data will be presented illustrating the superiority of this process over the customary manual method of tablet coating.

DESIGN AND EQUIPMENT

The schematic diagram in Fig. 1 illustrates the overall design and equipment involved in automating and programming the film-coating operation. Before detailing the various components of this process, a brief description of the operation and function of the programmer will be presented.

The coating process is controlled by a perforated

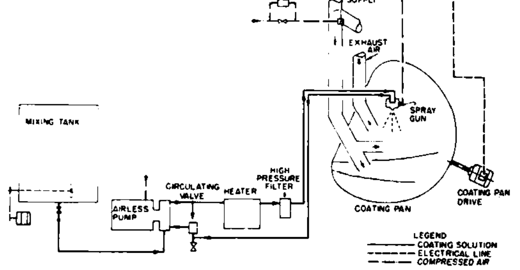

Fig. 1.—A schematic diagram of the design and equipment for the programmed automated coating operation.

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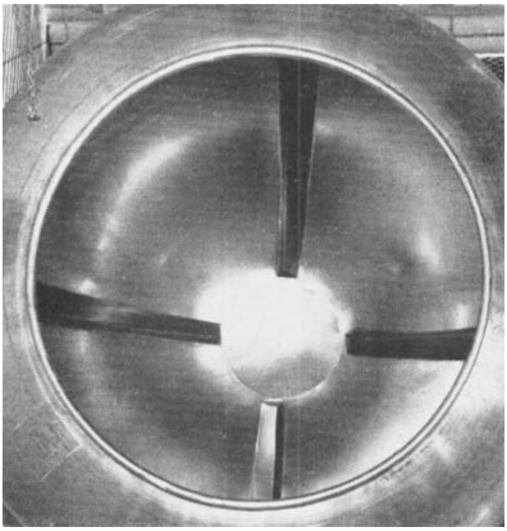

Fig. 2.—A photo depicting the baffle system.

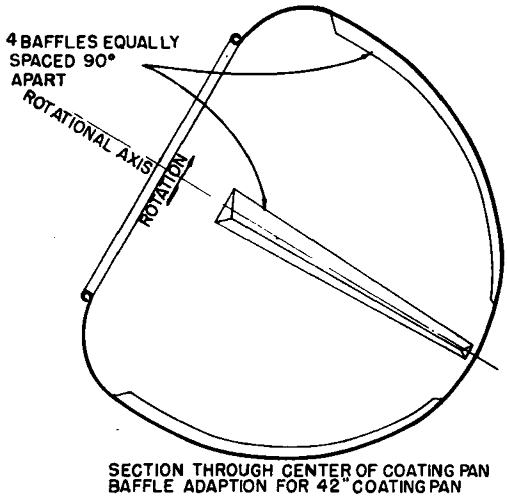

Fig. 3.—A schematic representation of the baffle design and placement in the coating pan.

tape traveling at a certain distance per unit time and transmitting its signals through a tape transmitter to the necessary relays in the programmer to activate the several components of the coating cycle, which include: (*a*) switching on coating-pan motor to start pan rotation; (*b*) regulating the pneumatic solenoid which opens and closes the automatic spray gun; (*c*) controlling a pneumatic solenoid which activates a single acting air cylinder that opens and closes the damper on the forced hot air duct; and (*d*) activating a latching relay terminating the coating operation.

In order to present a clear description of the design and equipment for this process, it has been divided into three segments which will now be discussed in detail.

Coating Pan, Forced Air, and Exhaust Ducts.—As mentioned earlier, the purpose of automating and programming the coating operation was to eliminate or reduce to a minimum any manual operations during the film-coating process. A phase of the coating operation where manual handling of the tablets is generally essential is subsequent to the addition of the coating solution. At this point the wet tablets tend to slide instead of tumbling. The coater usually places his hand into the pan and manually moves the tablets to aid the distribution of the coating solution until the tablets have partially dried and begin to tumble. In an effort to eliminate this manual handling phase of the coating operation, the influence of (*a*) pan rotational speed, (*b*) pan design, and (*c*) baffles placed into the pan on the tumbling action of tablets in the wet and dry state were investigated. Results from these tests indicated that the use of baffles of a particular design would provide the most suitable means for attaining this objective.

After considerable testing of different shapes, sizes, and numbers of baffles for use in a 42-in. pear-shaped stainless steel pan, the baffle system illustrated in Fig. 2 was found to be optimal. As is shown, the pan was divided into quadrants and a baffle was placed in each quadrant equidistant from one another. Figure 3 shows a schematic representation of the baffle designs and their placement in the coating pan. It is evident from this figure that the baffles have a gradual rise from the bottom to the top of the pan as well as slope in the direction of the pan's rotation.

The rise in baffle height from the inside to the outside of the coating pan was necessary to effect an even tumbling action throughout the pan, since the pan shape would cause the tablets to tumble at different speeds from the bottom to the top of the pan.

The slope of the baffles in the direction of the pan's rotation varies from 1 in. at the bottom to 2.5 in. at the top. This slope of the baffles is essential to prevent a too high carry over of tablets with a subsequent high fall causing irregular tumbling action and a possible focal point for tablet sticking and collection of coating solution and duster.

The baffles are 33 in. long, made of stainless steel, and fit the curvature of the pan. The baffles begin about 5 in. from the bottom center of the pan to about 10 in. from the top. Placing the baffles 5 in. from the center point of the pan gives a 10-in. diam. bottom area that is not baffled. This free area is necessary for correct tumbling action. The 10 in. at the top of the pan remain unbaffled for the same reason. If the baffles were extended at either end, the tablets no longer exhibit a smooth tumbling action, but rather a jumping action. It is also desirable to taper off both ends of the baffle gradually to enhance further smooth tumbling of the tablets.

Studies of the coating operation showed that sticking of tablets on the pan wall (when present) would radiate from the bottom center of the pan. Entering air is not sufficient to assist in drying this section of the pan wall because it is almost continuously covered with tablets. To eliminate this focal point for sticking, a Y attachment was developed for the forced hot air duct so that one part of the Y directed air to this focal point and the other part of the Y directed air onto the tablets. This is illustrated in Fig. 4 (the other duct shown in the pan is the exhaust duct which removes solvent vapors and dust).

Spray Equipment for Application of Coating Liquid and Duster.—With one of the human factors now eliminated, the other major variable was the

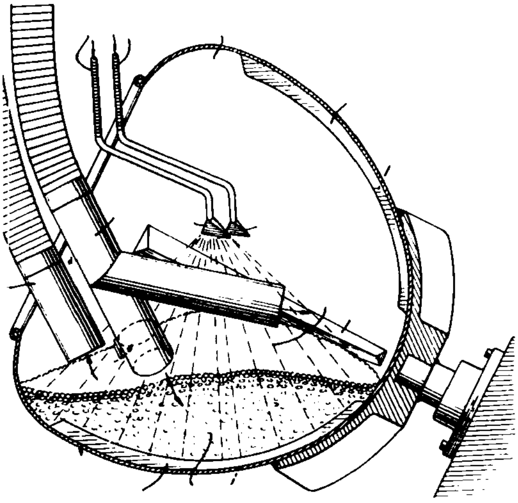

Fig. 4.—A schematic illustration of the Y attachment on the forced hot air duct.

application of the coating liquid and duster. In order to insure the addition of exact quantities of coating liquid and duster to the tablets at definite time intervals, this phase of the coating operation had to be placed on an automated basis and removed from the control of the tablet coater. To automate this operation, it was essential to develop an electronically controlled method of adding the coating liquid and duster. Spray guns for applying the coating liquid and duster seemed most feasible.

Existing methods for applying the coating liquid and duster to tablets consist of pouring a portion of coating liquid from a measuring container and a quantity of duster from a scoop. This method relies essentially on the tumbling action of the tablets for the distribution of coating liquid and duster. However, if the liquid and duster are added as a fine spray, the distribution of these coating materials on the tablets should be significantly improved compared to the hand method of addition. The reproducibility and uniformity of repeated coats can be accurately controlled through the use of a sprayed quantity of liquid and duster by regulating the duration of spray. This would in turn produce tablets exhibiting a more uniform and elegant film.

A spray process could be further improved by including the duster (*e.g.*, talc) in the coating liquid and applying the suspension as a spray, thereby eliminating the dusting step from the operation. This could produce four advantages: (*a*) talc in liquid suspension lends itself to easier fluidization than talc powder so that a reproducible quantity of material is sprayed per unit time; (*b*) using talc in liquid suspension permits a more uniform distribution of talc on the tablets being coated and consequently a thinner enteric film; (*c*) a reduction or elimination of the inherent dust problem of a fluidized talc spray resulting when talc in the form of a liquid suspension is used; and (*d*) the elimination of one operation from the coating cycle.

For film coating it seemed that a spray gun which gives an *airless spray* would be most desirable. The term *airless spray* is used to refer to a spray coating method in which a coating material is atomized by ejecting it from a spray nozzle by a hydraulic pressure imposed on the coating material itself.

The conventional *air spray method* atomizes the coating material by striking it with a high speed jet of compressed air at the end of the atomizing gun chamber. Because air is so light, it takes a tremendous amount of air moving at a high speed to atomize properly a viscous material. The stream emerging from an air atomizing gun is actually a high speed air current in which a small fraction of liquid is dispersed, generally about 1% or less. The liquid droplets are blown forward at high speeds and the atomizing air expands and rebounds causing much of the coating liquid to be blown away from the material to be coated. In addition, as the atomizing air expands, it also cools. This effect, coupled with the cooling effect of solvent evaporation, can lower the temperature of the atomized droplets far below the temperature of the surrounding air. If the temperature were to drop below the dew point, atmospheric water could condense on the particles and could cause the particles to deposit irregularly on the tablets, exhibit poor flow-out properties, cause the film to take on a blush appearance, and possibly rub off from the tablets.

However, the airless spray gun gives a completely different type of spray. Here, since no atomizing air is used, the coating material itself is discharged from the spray nozzle with possibly a minimum amount of air. The droplets move forward by their own momentum. The atmosphere between the spray gun and material to be coated resists the movement of droplets which are appreciably slowed down by this air resistance. Further, the energy released at the nozzle of an airless gun is only a fraction of that required in the air atomizing gun with a consequent drastic reduction in rebound or overspray from the material being coated. In addition, by the use of the airless spray gun, the amount of solvent vapors escaping from the coating pan is kept at a minimum and substantially below explosive and health limits. The airless spray gun would be particularly necessary if a duster were to be sprayed onto the tablets. One could just imagine the dust problem that would result if an air atomizing gun was used.

Figures 5 and 6 depict the relative spray characteristics for an air and airless spray gun using a coating liquid of the same viscosity. It is readily evident that a significantly greater overspray and rebound takes place for the air spray system as well as requiring a higher energy for atomization. Figure 6 shows that when the liquid is heated before spraying, a reduction of atomization energy takes place with the airless system requiring about one-third and the air system about two-thirds that of the cold spray.

Since it would be advantageous for the spraying system to be used for automating the coating process to have maximum versatility, a thorough investigation was made of the various spray equipment available. A unit of particular suitability and versatility was found in a Nordson airless spray-coating unit. This spray system atomizes the coating liquid, heated or unheated, by ejecting it from the spray nozzle through a hydraulic pressure imposed on the coating material itself.

The design and components of the spray system are schematically presented in Fig. 7. In this system the coating liquid in the supply tank is drawn up the siphon tube to the pump and pressurized, pushed

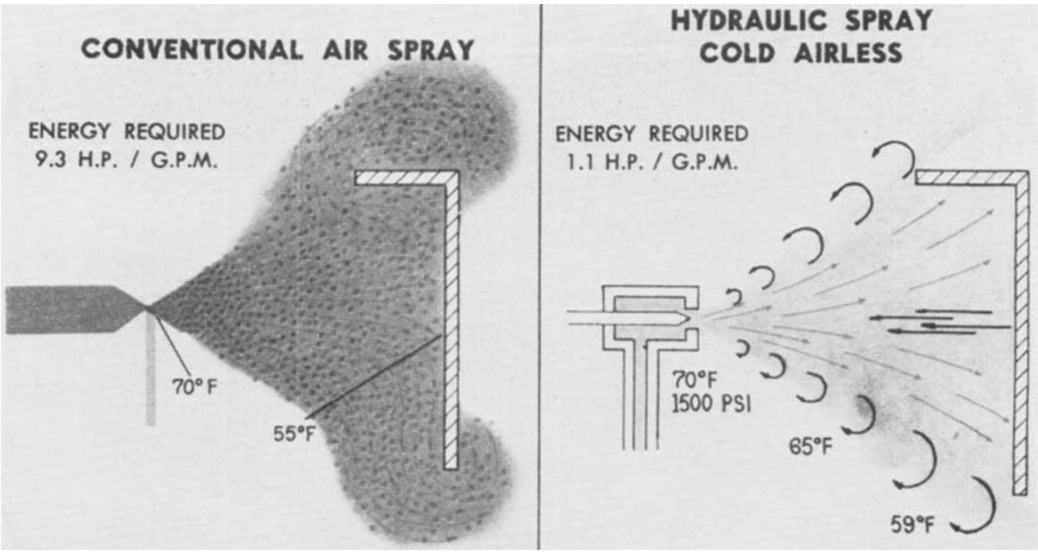

Fig. 5.—The relative spray characteristics of a liquid at room temperature for an air and airless spray system.

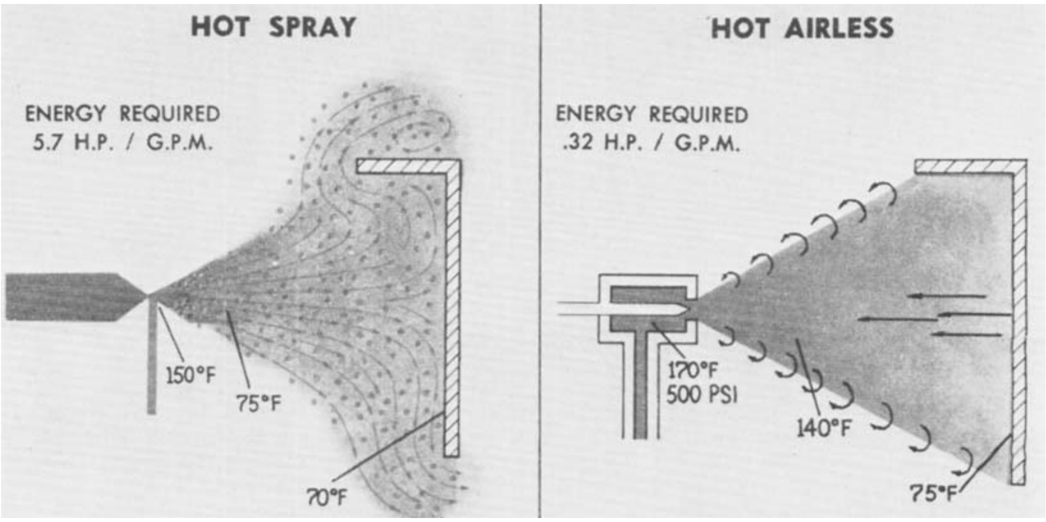

Fig. 6.—The influence of heated liquid on the spray characteristics of an air and airless system.

through the heater (which is specially designed to withstand the high hydraulic pressures needed for atomization), the high pressure filter (which removes particles in liquid suspension that are greater than the nozzle orifice), and then flows to the spray guns available for atomization. The Nordson automatic guns are of a special design making it possible to open and close the gun in less than *one-hundredth* of a second.

This spray system is designed to keep the liquid in the unit circulating to and from the gun when the gun is and is not spraying. When the system is not spraying, the coating liquid travels from the gun through a return line and then through a recirculating valve permitting a controlled amount of liquid to pass into the suction part of the pump. From here the liquid is repressurized for circulating through the system. This assures a continual flow of material through the system; the coating material at the nozzle is always at the correct temperature and ready to be sprayed whenever the gun is triggered. In addition, it prevents material in suspension from settling out as well as eliminating possible clogging of the orifice tip due to particle accumulation resulting from the loss of volatile solvents. When the gun is triggered and coating liquid forced out of the nozzle, a similar amount of

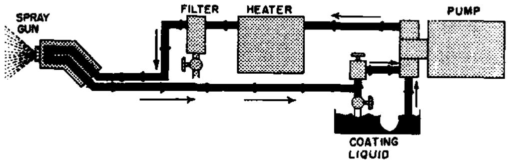

Fig. 7.—Design and components of the airless spray system.

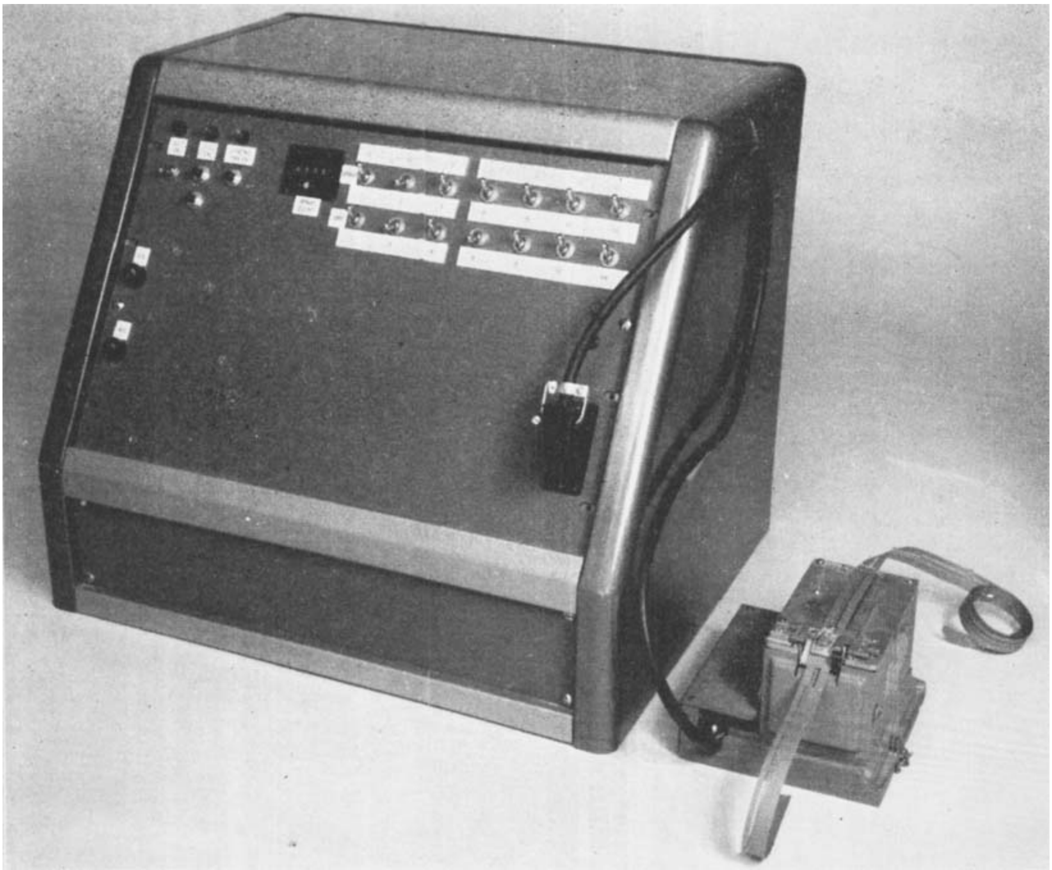

Fig. 8.—Photo of programmer used to automate coating operation.

liquid is withdrawn from the supply container and added to the recirculating pump. Therefore, recirculation is maintained even when the system is spraying. In this system coating liquid does not flow back into the supply container. If it did, then the entire supply of coating material would eventually become heated causing evaporation of the low boiling solvents in the coating material.

In our studies, carbide tip nozzles giving a flat spray pattern were used. The flow rate of the liquid through the nozzle is dependent upon the viscosity and temperature of the liquid, hydraulic pressure, and orifice measurements. However, the major controlling factor for flow rate and spray angle is the nozzle geometry and dimensions.

Automation and Programming of Film-Coating Operation.—With the spray equipment chosen and the coating pan modified to permit tablets to tumble when wet or dry, the final step was to design a programmer to automate the coating operation and remove the human element completely from the coating process. After studying various ways to program the film-coating operation, a punched tape traveling at a certain distance per unit time and transmitting its signals through a tape transmitter to the necessary relays to activate the several elements of the coating cycle appeared to be the simplest to design, install, and operate. In addition, such a system could be built with binary circuitry giving maximum flexibility relative to making changes in the time allotted to each sequence of the cycle. When the changes in cycle time involve only the repetitive spray-dry cycles of the coating operation, the same tape can be employed and the changes made on the programmer by adjusting the toggle switches controlling the binary circuitry. Only when the irregular parts of the coating cycle require changing is it necessary to use a new tape. Therefore, such a programming system can readily be used for various tablet formulations requiring different coating cycles. Any programming arrangement can be obtained with this system by modifying the tape or modifying the binary circuitry on the programmer. Paper tapes can generally be used between 50 to 100 times before replacement. If greater permanency is desired, mylar tapes can be used.

Figure 8 shows the programmer which consists of a Western Union tape transmitter designed for 5-unit code transmission using perforated tape with in line feed holes. The perforated tape travels at a certain distance per unit time transmitting its signals through the tape transmitter to the necessary relays in the control box which activates the several elements of the coating cycle. The timer in the control box regulates the residence time in the transmitter for each hole in the tape.

As the tape is pulled over the code pins of the tape transmitter by the feed wheel traveling at a certain rate, signals are transmitted to the relays in the control box activating the various functions of the coating cycle and (*a*) starts coating pan rotation by switching on motor through a relay and step down transformer; (*b*) regulates the three-way pneumatic solenoid which opens and closes the automatic spray gun used to spray doses of coating suspension; (*c*) controls a pneumatic solenoid which activates a single acting air cylinder which opens and closes the damper to the forced hot air duct, supplying the hot air for drying the tablets; and (*d*) activates a latching relay which terminates the coating cycle and shuts off the programmer.

By employing this programmed automated process of tablet coating, the volume of coating suspension and, if needed, the weight of talc can be controlled by the length of time of spray at constant hydraulic pressure and nozzle size. Consequently, it should be possible to obtain readily reproducible coatings from batch to batch of tablets.

EXPERIMENTAL

The influence of the human factor on the disintegration properties, uniformity, and physical appearance of enteric films will be illustrated with data obtained from coating $^{15}/_{32}$-in. modified ball tablets weighing 1.06 Gm. by both the customary coating techniques and by the programmed automated process. In both instances the coating operation was performed on 85 Kg. batches of tablets in the stainless steel 42-in. pear-shaped baffled coating pans. For the automated process the tablets were coated with a suspension of 10% talc in a 10% solution of cellulose acetate phthalate containing diethyl phthalate as plasticizer and a solvent system of anhydrous ethanol-acetone, while for the customary technique the same coating solution was used except that the talc was added as a duster between coats instead of in suspension.

In the automated process the suspension was sprayed at a temperature of 50° and at a flow rate of about 1000 ml./minute. The hot air flowing into the pan to dry each coat was at 47°. The coating cycle employed was (*a*) an initial 90-second spray and 120-second dry period; (*b*) then repetitive cycles of 15 seconds consisting of 3-second spray and 12-second drying periods; (*c*) at the end of the repetitive cycles, a 5-minute drying period is permitted in the pan as the tablets tumble; (*d*) then the tablets are placed in an oven at 40° for thermosetting of the film coating and removal of residual solvents.

The normal coating procedure is the term applied to the procedure that was used in our Production Division and consisted of (*a*) manually spraying a quantity of coating solution onto the tablets, (*b*) permitting the tablets to tumble for a prescribed period of time, (*c*) manually dusting on a quantity of talc, (*d*) allowing the tablets to tumble to permit distribution of the talc, and (*e*) directing forced heated air onto the tablets for drying the coatings. These steps are repeated until the desired coating is obtained. At the completion of the coating operation the tablets are removed from the coating pan and placed into a circulating-air oven maintained at 40° to thermoset the film.

By removing samples of tablets during the programmed automated coating operation it was found that the tablets develop enteric properties between 150–200 repetitive cycles. Additional coatings within definite limits provide a safety margin for disintegration properties and improved appearance. Table I shows comparative data of film weight and film uniformity from batch to batch of tablets prepared by the automated process after 200 and 350 repetitive cycles (called end coating) and tablets prepared by customary coating techniques. It is readily evident from this table that it is possible to obtain enteric film coated tablets by the automated spray process with film coatings of about one-third the weight of those prepared by customary coating techniques. In addition, the range of mean coating weights for the 200 and 350 repetitive cycle (called end coating) coated tablets are approximately one-fourteenth and one-sixth that of tablets coated by customary manual coating techniques.

TABLE I.—MEAN WEIGHTS OF COATING OBTAINED BY AUTOMATED AND NORMAL COATING TECHNIQUES

	Mean Coating Weights	
Batches	200 Coats	End Coating
Normal A	...	0.1007
Normal B	...	0.0914
Normal C	...	0.0736
Normal D	...	0.1269
Overall Mean	...	(0.0981)
Range of Means	...	0.0533
Automated A	0.0358	0.0594
Automated B	0.0341	0.0639
Automated C	0.0365	0.0580
Automated D	0.0378	0.0671
Automated E	0.0370	0.0639
Overall Mean	(0.0362)	(0.0623)
Range of Means	0.0037	0.0091

Table II summarizes the standard deviations for the uncoated and coated tablets used for the normal and automated processes. Comparing the overall mean standard deviation of uncoated tablets used for the normal and automated process of coating, it was found that they do not differ significantly from one another. However, in comparing the overall mean standard deviation for the tablets coated by the normal techniques with those coated by the automated process it is found that the overall standard deviation for the tablets coated by the automated process is significantly less than that for the tablets coated by the normal process.

TABLE II.—STANDARD DEVIATIONS OF MEAN WEIGHTS OF UNCOATED AND COATED TABLETS

Batches	Uncoated Std. Dev.	End Coating Std. Dev.
Normal A	0.0050	0.0203
Normal B	0.0064	0.0222
Normal C	0.0091	0.0288
Normal D	0.1110	0.0315
Overall Mean	(0.0079)	(0.0257)
Automated A	0.0106	0.0098
Automated B	0.0106	0.0130
Automated C	0.0106	0.0100
Automated D	0.0102	0.0095
Automated E	0.0059	0.0100
Overall Mean	(0.0089)	(0.0105)

The time required for the automated process to give tablets coated by 200 repetitive cycles is about 60 minutes and by 350 repetitive cycles, about 90 minutes.

From our experiences with this programmed automated process of coating it became readily apparent that it offered a number of advantages over the manual method of coating, the most significant ones being:

Tablets from a particular pan batch show uniform coatings relative to physical appearance and disintegrating properties.

Reproducibility of coating from batch to batch of tablets is insured.

The more even film coating deposited by this process produces enteric coated tablets with one-third or less the film weight necessary for manual coating procedures.

Using this process with the repetitive short spray-dry cycles of coating, no coating build-up on the pan wall takes place. This permits the continuous coating of batches of tablets without the need of washing the pans between batches.

The possibility of pimples forming on the coating is eliminated because no coating build-up on the pan wall results.

The time required to obtain enteric film coated tablets by the automated process is at least half that required for the manual process.

A manifold increase in production capacity is possible through this process of film coating.

One operator will be able to supervise the coating of many batches of tablets per day.

It is unnecessary to use an experienced tablet coater with this system of film coating.

This method of coating is adaptable for use with water-soluble films as well as sugar coating.

SUMMARY

A programmed automated process for film coating has been developed which is capable of concurrently coating multiple pan batches of tablets containing 85–100 Kg. of tablets per pan.

As a result of eliminating the human element from the coating operation and consequently the so-called "art" of tablet coating, film-coated tablets prepared by the automated process show close reproducibility of coating from batch to batch of tablets as well as greater uniformity of coating within batches of tablets.

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Notes

Concentric Double Electrolytic Junction Calomel Reference Electrode

By NORMAN ADLER

Aqueous calomel reference electrodes are frequently unsuitable for use in titrations in acetic acid or acetic anhydride as they often lead to erratic potentials and drifting, irreproducible end points (1–4). These difficulties have been attributed to contamination of the electrode (3), to insufficient electrode-solution interfacial contact (1), and to changes in the liquid junction potential as the electrolyte flows from the electrode to the solution (4). Asbestos fiber-type electrodes, notoriously troublesome in nonaqueous titrations (4, 5), may be particularly prone to contamination. As the titration solution (often containing perchlorate ion) diffuses into the fiber, it mixes with saturated aqueous potassium chloride. Less soluble potassium perchlorate may then be precipitated, thereby disturbing or breaking the electrolytic contact. Depending on the titration medium, the electrolyte may also be precipitated. A crust of precipitate has been observed to form at the tip of the fiber in contact with the titration medium (6). Fiber electrodes that become shorted or erratic during nonaqueous titrations may often be rejuvenated by soaking overnight in water.

The difficulties with aqueous electrodes may also be attributed to the nature of the electrolyte and the solvent used. The addition of water to the titration medium changes the nature of the system, often deleteriously (3). Water and the occasionally used methanol (7, 8) are not suitable for acetic anhydride systems as they undergo acid-catalyzed

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