coating process should there be a failure in one component of the systems such as in the spray delivery timer. Even though timed devices may contain fail-safe mechanisms, such a timed coating process would have to be terminated and reprogrammed manually.

SUMMARY

An automated tablet-coating system was designed which is self-programming and based on the rate of moisture loss. The Moisture Analyzer, which programmed the system, was revealed to be extremely sensitive to the presence of moisture and to moisture loss in the tablet mass. The system quickly adjusts to any change in spray cycle time and also shows a high degree of reproducibility between cycles under similar conditions.

A high degree of sensitivity of the drying cycles to changes in the environmental humidity was also demonstrated by the apparatus. It is, therefore, felt that a moisture-sensing device should be utilized when considering an automated tablet-coating system for production.

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Monitoring Volatile Coating Solution Applications in a Coating Pan

RUSSELL J. LANTZ, Jr.*, ALAN BAILEY, and MANFORD J. ROBINSON

Abstract ☐ Equipment and methods are presented for recording temperature patterns resulting from the evaporation of volatile coating solutions applied to pellets in a rotating pan. These patterns are interpreted in regard to run-to-run replication, effect of application and drying rate, and coating solution distribution. The applicability of this equipment and methodology to control the coating of solid particles is discussed.

Keyphrases ☐ Coating application, pellets—monitoring method ☐ Volatile coating solutions—application monitoring ☐ Diagram—pellet coating equipment with monitor ☐ Thermal patterns—pellet coating

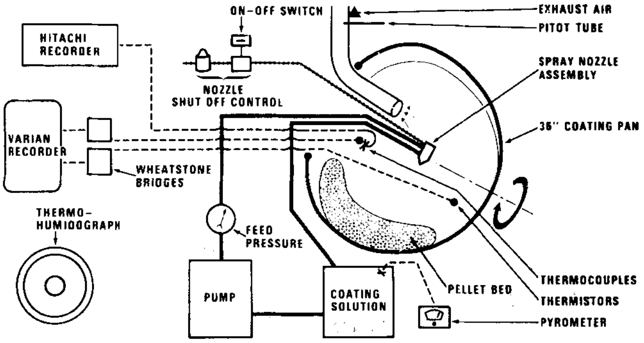

Figure 1—*Equipment diagram.*

Sutaria (1) has noted that, until recently, pharmaceutical coating processes were an art because of the apathy shown toward studying the many variables involved in these processes. Sutaria's extensive bibliography indicates that efforts to define and control these variables are now underway and, in some cases, have resulted in the semiautomation of certain aspects of the coating process.

The purpose of this preliminary report is to add to this knowledge by presenting data on methods found useful for monitoring the application of volatile coating solutions to pellets in a rotating pan.

The application and evaporation of coating solutions containing volatile solvents, *e.g.*, acetone, alcohol, and chloroform, produce measurable temperature changes in a bed of pellets in a rotating pan. By placing a thermocouple and thermistors in the pellet bed and recording the temperature changes during coating, it is possible to discern and control certain events and/or trends taking place during the coating process.

EXPERIMENTAL

Equipment and Instrumentation—The coating process was performed in a Stokes 91.4-cm. (36-in.) coating pan.[1] The volume of air moving across and through the pellet bed was monitored at the exhaust with a Dwyer pitot tube and an air velocity meter No. 400.[2] The temperature of the coating solutions was controlled in a 30-l., electrically heated, feed tank. The feed tank temperature was monitored with an Alnor pyrometer[3] coupled to a standard

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[1] Model 900-1-8, Pennwalt Corp., Stokes Tableting Equipment Dept., Warminister, Pa.

[2] Dwyer Manufacturing Co., Michigan City, Ind.

[3] Type 1200, Illinois Testing Laboratories, Inc., Chicago, Ill.

Table I—Data Sheet Sample of Conditions Recorded in Addition to Thermal Patterns

Code 20 Time	Pellet-Bed Temp.	Exhaust Air Flow, c.f.m.	Ambient Conditions Dry Bulb Temp.	Ambient Conditions % Relative Humidity	Pump Operation and Coating Solution Application Pump Driving Air, psig.	Pump Liquid Pressure, psig.	Coating Solution Applied per Hour, kg.	Coating Solution Spray Feed Rate, g./min.	Coating Solution Temp.
10:00 a.m.	15°	200	27.7° (82° F)	31	10	130	4.9	354	67°
11:00 a.m.	15°	211	27.7° (82° F)	31	10	130	4.9	346	68°
12:00 noon	14.5°	211	26.6° (80° F)	33	10	130	4.9	354	64°
1:00 p.m.	14.5°	211	26.6° (80° F)	31	10	130	4.9	344	64°
2:00 p.m.	14.5°	199	26.1° (79° F)	34	10	130	4.9	356	63°
3:00 p.m.	14.5°	200	26.1° (79° F)	34	10	130	4.9	358	65°
4:00 p.m.	15.5°	205	26.1° (79° F)	34	10	125	4.6	355	64°

Table II—Program for Coating Solution Application and Pellet Drying

Code 20 Time	No. of Cycles	Spray Cycle, min.	Dry Cycle, min.	Total Spraying per Hour, min.	Total Amount of Coating Solution Applied per Hour, kg. (Theory)
1st hour	5	2	2	10	3.50
	10	1	3	10	3.50
2nd hour	15	1	3	15	5.25
3rd and 4th hours	15	1	3	15	5.25
5th hour	12	1	4	12	4.2

iron-constantan thermocouple. The coating solutions were pumped by a Grover pump,[4] which delivered 130 psig. pressure to a No. 650067 Spraying Systems[5] T-Jet nozzle mounted on a Spraying Systems 24 AU Auto Jet valve assembly.[5] The spraying and drying cycles were timed with a stopwatch, and the "start" and "stop" of the spray nozzle were controlled by an on-off switch in series with a Skinner electric valve.[6] Ambient conditions were recorded with a Bristol thermo-humidograph.[7]

An Hitachi 165 recorder[8] fitted with a standard iron-constantan thermocouple, placed 3.8 cm. (1.5 in.) deep in the pellet bed and 17.9 cm. (7 in.) in from the pan rim, was used to record pellet bed temperature at this point. The recorder temperature range was from 4 to 21°.

A second recorder,[9] with two type A-21 amplifiers, was used to amplify the signals from two thermistors[10] (nominal resistance: 2000 ohms at 25°). To obtain a range of 13–16° with each amplifier, the thermistors were placed in one arm of a standard Wheatstone bridge, where all arms were at equal resistance at 14°. One thermistor was placed beside the iron-constantan thermocouple, while the second thermistor was moved to different points on the pellet-bed surface. The two separate recording setups were used to give the advantage of the wide temperature range coverage of one recorder and the detailed or expanded scale of a small temperature range recorder, and to provide a reference check of one recorder against the other.

Figure 1 shows the equipment setup.

Materials—Sugar pellets, 16–30 mesh, were used for each coating run. Special denatured No. 30 alcohol was the volatile solvent used in preparing the coating solution.

Procedure—For each coating run, a 50-kg. charge of pellets was placed in the coating pan preset to rotate at 20 r.p.m. The coating solution was prepared, heated, and maintained at 65 ± 3°. A 2-min. spray and 2-min. dry cycle was used until the pellets were wetted and the bed temperature dropped to approximately 15°. The time cycle was then changed to a 1-min. spray and a 3-min. drying period, or a 1-min. spray and a 4-min. drying period, to maintain an equilibrium between the amount of coating solution applied and the rate of evaporation. A sample data sheet and program for coating solution application and pellet drying are seen in Tables I and II, respectively.

Some coating runs were made under near identical conditions to determine if identical thermal patterns could be obtained. In other runs, coating application rates and pellet drying times were varied. The temperature profile across the pellet bed and the circulation of coating solution to the back of the coating pan were also determined. Sixteen coating runs were made to accumulate and recheck the data obtained.

RESULTS AND DISCUSSION

Typical thermal patterns, taken at the same point in the pellet bed with the thermocouple and thermistor setups, are shown in Fig. 2. These identical curves show good uniformity from cycle to cycle in the temperature rise that accompanies the heated coating solution application and the evaporative cooling which occurs during the drying period. The range between the maximum and minimum temperature usually averaged 0.3–0.4° within a normal spray and dry cycle (the lower curve).

Coating runs performed on different days, following nearly identical conditions and procedures, show almost identical thermal traces throughout each run. This is illustrated in Fig. 3, where portions of the trace of each run are shown early and late in the coating process. A comparison of the thermal patterns made early in the run with those made late in the run shows a difference in the heating portion of the curve, which represents the coating solution application. It is possible that the leveling off or temperature decrease at one point in the heating curve may be caused by a partial dissolution of the heavier coating accumulated late in the run as new coating solution is applied. This effect is shown more clearly in an expanded thermal trace of one cycle in Fig. 4.

It was found that a variation in coating solution application rates and/or pellet drying times caused distinguishable differences in the

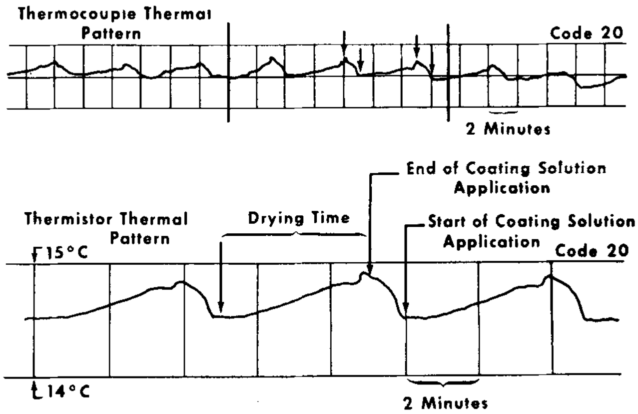

Figure 2—*Typical patterns obtained with thermocouple and thermistor probes.*

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[4] Model 2585 TSS, Grover, Montebello, Calif.
[5] Spraying Systems Co., Bellwood, Ill.
[6] Skinner Electric Valve Division, New Britain, Conn.
[7] Bristol Co., Waterbury, Conn.
[8] Hitachi Ltd., Tokyo, Japan.
[9] Model G22, Varian Associates, Palo Alto, Calif.
[10] Fenwal type GB 32 P8, Fenwal Electronics, Inc., Framingham, Mass.

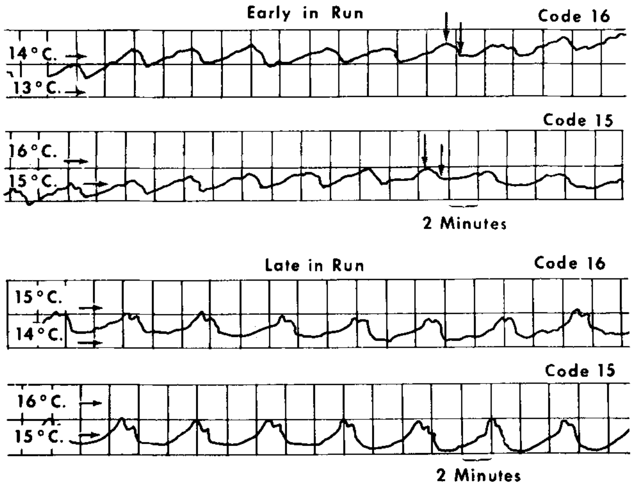

Figure 3—*Run-to-run duplication of thermal patterns.*

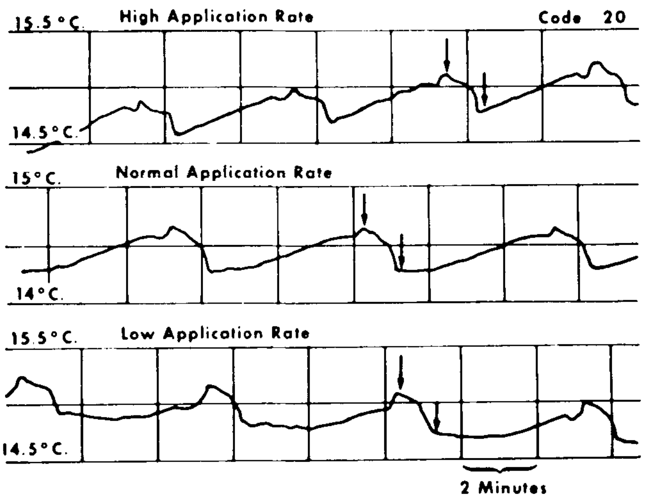

Figure 5—*Effects of coating solution application rate and drying.*

pellet-bed thermal patterns. In Fig. 5 the upper and lower thermal traces show the effect of high and low coating solution application rates, and the middle thermal trace represents a more normal application and drying rate. High application rates of coating solution result in a continuous decrease in temperature during drying, with an overall downward trend in pellet-bed temperature; the low application rate of coating solution shows just the opposite effect. The normal application rate curve illustrates a system in equilibrium, in which the coating solution solvent application rate balances the solvent evaporation rate with no upward or downward trend in pellet-bed temperature.

If the pellets are subjected to an extended period of drying, or if only small amounts of coating solution are applied, two stages of drying, namely the "constant rate period" and the "falling rate period" (2, 3), are revealed by the thermal traces of these pellet beds. In the constant rate period, unbound solvent is removed from a saturated surface at a constant rate under constant conditions. The second stage or falling rate period proceeds after the critical solvent level has been reached,[11] and it is controlled by the internal movement of the solvent to the surface of the pellets. This stage is characterized by a diminishing solvent evaporation rate under constant conditions. In Fig. 6, the constant rate period is shown as a constant decrease in pellet-bed temperature, while the second stage or falling rate period is shown as a gradual increase in pellet-bed temperature.

The distribution of coating solution in a rotating pan is always of concern, because of the poor product circulation patterns set up in some coating pans, including those with improper baffle designs. Improper positioning of the spray nozzle can also result in maldistribution of the coating solution. A means of evaluating coating solution distribution in a rotating pellet bed is illustrated in Figs. 7–9. Figure 8 shows four pellet-bed surface thermal patterns taken at points shown in Fig. 7. Assuming that the magnitude of pellet-bed temperature change is an indication of the amount of coating solution present, it is evident that Position B, just forward of the nozzle, E, receives the most coating solution, followed by Position C just behind the nozzle. Position A receives the third highest quantity of coating solution, while Position D, at the back of the coating pan, receives the least amount. Thermal patterns in Fig. 9, taken at the rear of this particular coating pan in Position D (Fig. 7), indicate that the amount of coating solution reaching this area depends in part on the coating solution application rate.

Measurement of temperature changes should be applicable to any pan coating process (including the coating of pellets and granules and the film coating of tablets) when coatings containing volatile solvents are used. Records of thermal patterns may be particularly useful in determining the day-to-day variability of one coating operator and/or the operator-to-operator variability for a single coating process.

Information which could reduce operator variability can be obtained, in part, from such a series of thermal pattern recordings. This technique may also turn out to be useful in coating process automation. Recording the thermal changes on magnetic tape and running a "playback" of the tape with proper circuitry and control[12] should permit automatic regulation of the pan coating process.

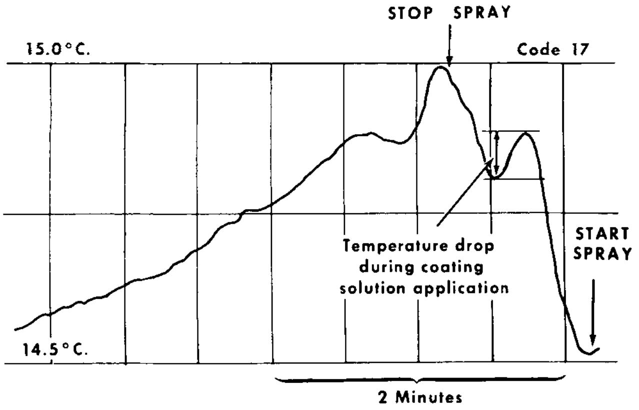

Figure 4—*Temperature drop during coating solution application.*

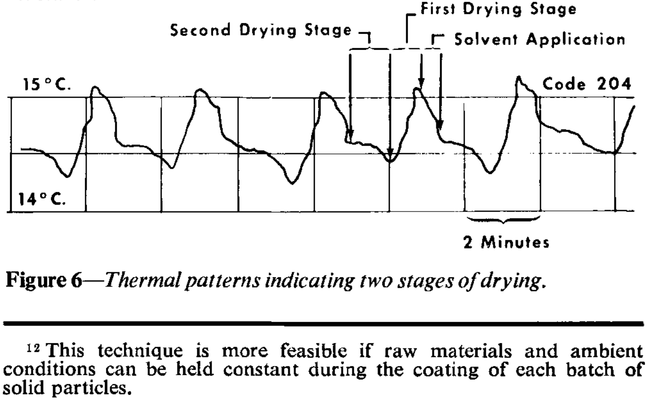

Figure 6—*Thermal patterns indicating two stages of drying.*

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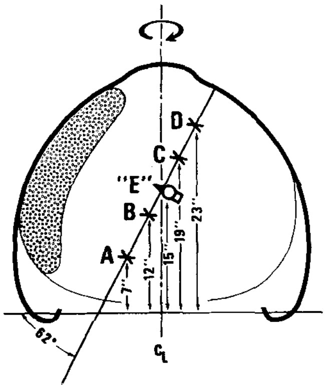
[11] The point where the surface no longer remains saturated.

[12] This technique is more feasible if raw materials and ambient conditions can be held constant during the coating of each batch of solid particles.



To the left is a TOPVIEW of the coating pan
Thermistor positions marked A, B, C, & D
are surface readings.

The spray nozzle "E" is positioned 4 inches
above the pellets and the nozzle axis is
perpendicular to the plane of the pellet surface.

Figure 7—*Nozzle and thermistor positions in the coating pan.*

The recording of temperature profiles across a bed of solid particles during coating may also be correlated with final product results to help in evaluating the design of coating pans and/or baffles for a particular product. This same technique should also be useful in selecting the proper spray nozzle design and determining its position in a coating pan.

SUMMARY

This preliminary report describes a method for measuring temperature changes encountered during the application of volatile coating solutions to pellets in a rotating pan. These temperature changes develop characteristic patterns during the periods of coating solution application and pellet drying, and these patterns are replicable. Distinguishable differences in thermal patterns were found when coating solution application rates and pellet drying times were varied. Thermal traces from extended pellet drying times revealed two stages of drying. Temperature changes recorded at several points on the pellet-bed surface demonstrated differences in coating solution distribution to the pellets.

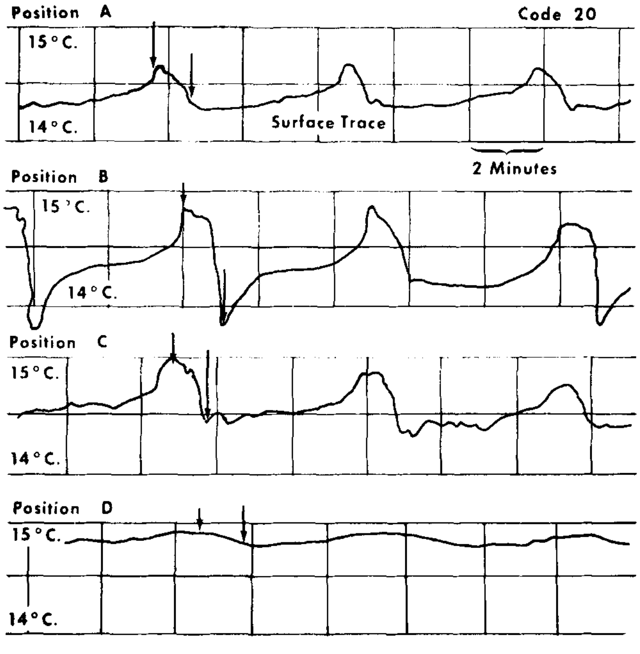


Figure 8—*Distribution of coating solution in the pellet bed.*

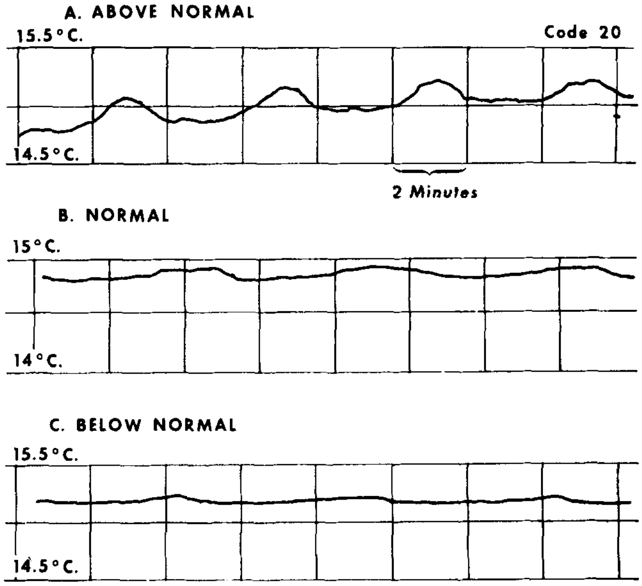


Figure 9—*Coating solution circulation to the back of the coating pan.*

This method of measuring solid particle-bed temperature changes in a rotating pan should be applicable to the control of day-to-day and operator-to-operator variability; to the automation of the coating process; and to an evaluation of the design of coating pans, baffles, and auxiliary equipment.

Correlations between changes in pan coating conditions and their effects on final product characteristics using this technique will be considered in another paper.

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