Improved Self-Programming Automated Tablet-Coating System

ALLEN HEYD and JOSEPH L. KANIG

Abstract ☐ The usefulness of a monitoring system based on moisture sensing is established as a means of automating a tablet-coating process. A radio frequency absorption system was employed to measure continuously the state of dryness of the tablet mass and to indicate when the tablets reached a predetermined satisfactory end-point. The moisture-sensing apparatus was found to monitor the drying rate accurately after the tablets had been sprayed. The analyzer readings were shown to be directly related to conditions of relative humidity existing in the environment surrounding the coating pan. The instrument was extremely sensitive and permitted a constant readout of drying rate and moisture content by means of a specially constructed probe placed within the tablet mass.

Keyphrases ☐ Tablet-coating system—self-programming, automated ☐ Automated tablet coating—radio frequency system, moisture measurement ☐ Diagram—automated tablet-coating system ☐ Moisture analysis—tablet-coating rate control

One important factor in a tablet-coating process is the drying of the tablet mass after each application of a coating solution. The rate of liquid loss dictates the time period between tablet-coat applications. One does not know if the rate of liquid loss is constant for each application during a coating process, or if the application of successive coats lengthens or shortens the drying time. It becomes a matter of subjective evaluation to determine whether or not a tablet mass is sufficiently dry before the next application. When an automatic timed procedure is used, it is based on the assumption that the drying rate is constant for each cycle of the process. The validity of this assumption has never been clearly established. Drying rates are different for each type of coating solution and are also influenced by tablet shape, tablet size, and batch size. A contention underlying this investigation was that an automatic timed procedure does not take into account either inherent or environmental conditions which may exercise an effect during the coating process and may vary the drying time. In taking these inherent or environmental changes into account, a system must be able to monitor the drying rate continuously and utilize the degree of dryness for controlling an automated process.

Automated tablet-coating systems that have been suggested to date are time controlled (1–6) with the length of each step in the coating cycle determined by a preset timer. Therefore, such systems are insensitive to environmental changes. When an automated system is created around a timed procedure, it must be programmed specifically for all the variables indicative of the properties of a particular batch. Each major variation in size, shape, weight, number, and other features of a tablet batch may require a separate and different program. A system that follows the drying rate, however, may prove to be independent of most of these variables. This report presents a description of an automated tablet-coating system which is entirely programmed and controlled by the moisture content of the tablet mass.

EXPERIMENTAL

Materials—The core tablets used in this investigation consisted of dicalcium phosphate dihydrate[1] lubricated with 1.5% magnesium stearate.[2] Tablets weighing 400 mg. were compressed with 1.01-cm. (0.40-in.) standard concave punches and used without further treatment.

The coating solution used in all the experiments was an 85% (w/v) syrup solution. The solution was colored with 0.1% (w/v) amaranth.[3]

Equipment—The schematic diagram in Fig. 1 illustrates the overall design and arrangement of the equipment which made up the programmed automated tablet-coating system.

The coating solutions were delivered by an airless spray system (A).[4] The pump model used (the "President") had a 28:1 pressure ratio. Two spray guns (B) (Graco automatic Hydra-Spray guns, model 205-163) were suspended inside a 96.52-cm. (38-in.) baffled coating pan (C) in such a position that the spray was directed perpendicular to the surface of the tumbling tablets for maximum, uniform coverage. The guns were suspended from steel tubing which was mounted on the pump chassis. A high-pressure fluid heater (D) (Graco model 206-580, series A), 240 v., was mounted on the rear of the spray system and connected to the fluid lines between the pump and the spray guns. The fluid passing through the guns could then be heated to a desired temperature before it was sprayed. A circulating system (E) (Graco Restrict-A-Flo) was utilized to accomplish continuous fluid circulation for maintaining uniform temperature and homogeneity of the coating liquid. Two timers[5] (F) were mounted on the automatic spray-system control panel. These timers were connected to energize the spray-on and drying cycle. The dry cycle timer was utilized only when the system was manually controlled. The timers were also interconnected with the moisture monitoring–programming device so the entire process could be evaluated for its applicability to automation. The instrument used to measure relative humidity during the coating experiments was a wet-dry bulb psychrometer.[6] Exhaust (G) was supplied with a portable vacuum,[7] and a blower-heater system[8] was used as the source of forced air (H).

---

[1] Stauffer Chemical Co., Chicago, Ill.
[2] Ruger Chemical Co. Inc., Irvington-on-Hudson, N. Y.
[3] National Aniline Div., Allied Chemical Corp., New York, N. Y.
[4] Graco Hydra-Spray Unit, Gray Co., Inc., Minneapolis, Minn.
[5] Microflex, Eagle Signal Corp., Moline, Ill.
[6] Psychron, model 566-3, Bendix Aviation Corp., Friez Instrument Div., Baltimore, Md.
[7] Craftool model 411, Craftool Inc., New York, N. Y.
[8] Model 7866, Bonat Inc., Norwalk, Conn.

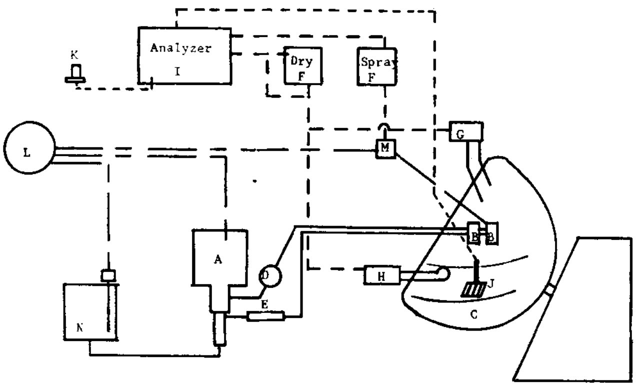

Figure 1—*A schematic diagram of the design and equipment for a self-programming automated tablet-coating system. Key: A, airless spray system; B, spray guns; C, coating pan; D, fluid heater; E, circulating system; F, timers; G, exhaust; H, forced air system; I, Moisture Analyzer; J–K, electrodes, automatic and manual, respectively; L, compressed air; M, solenoid switch; N, mixing tank;* ——, *coating solution;* - - -, *electrical line; and* — - —, *compressed air.*

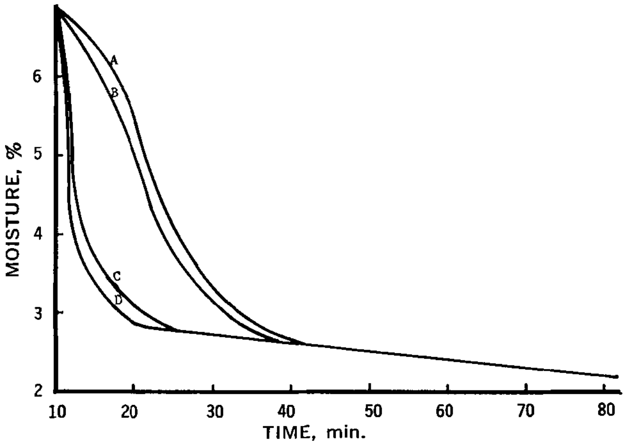

Figure 3—*Analyzer sensitivity to moisture loss. Key: A, at 29°; B, at 29° with exhaust; C, at 29° with forced air and exhaust; and D, with forced air at 50° and exhaust.*

The instrument selected to measure the state of dryness of the tablet mass was the HFR-6E Moisture Analyzer.[9] The Analyzer (I) operates on the principle of high-frequency conductance caused by power absorption of the material being tested. Power absorption or conductance is the only electrical property which gives accurate indications of moisture content (7). The Analyzer was adapted for in-process control of the various components of the coating system and for continuous readout of tablet moisture content. This was accomplished by replacement of the regular conductance meter with a contact-conductance meter which energized the spray cycle timer. Auxiliary switches were also incorporated into the Analyzer which activated the blower-heater and the exhaust system at the proper coating cycle intervals. A digital contact meter on the Analyzer was set to predetermine the total number of coats applied, after which it deenergized the entire coating apparatus. L-shaped and cup-shaped electrodes (J, K) were used to determine sample moisture content automatically and manually, respectively. Analyzer meter readings did not significantly differ when using either electrode under the same set of conditions. The cup electrode was used only during calibration curve preparation.

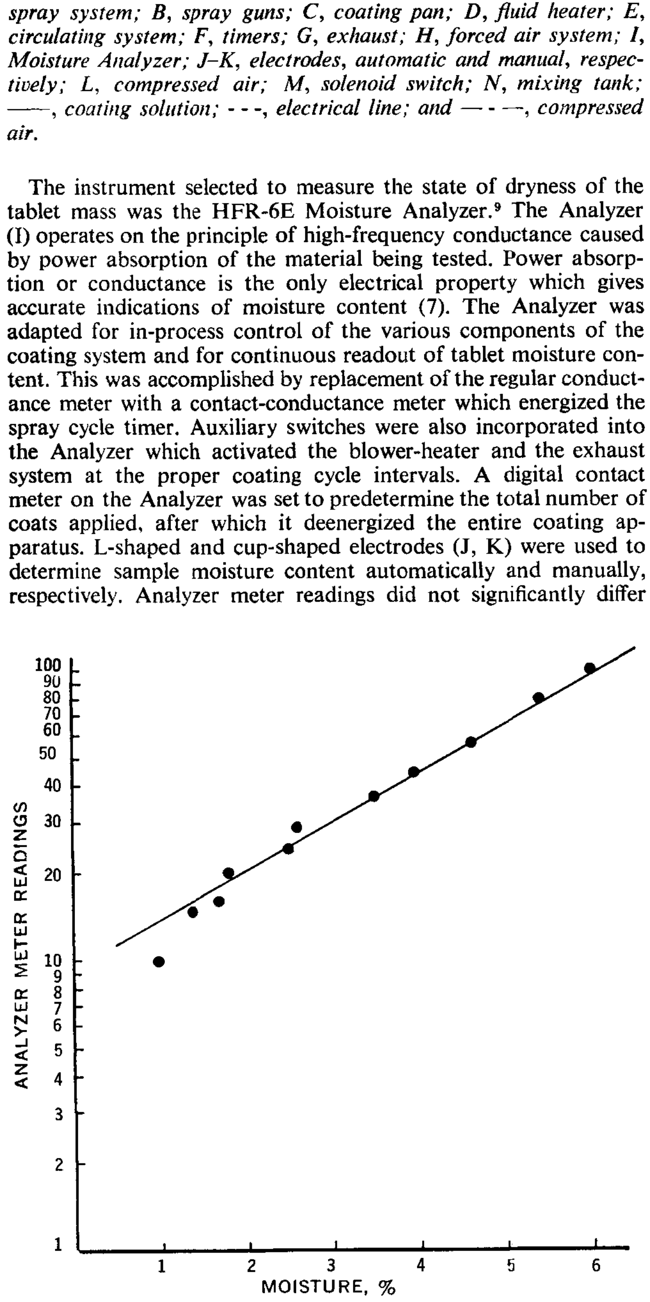

Figure 2—*Moisture calibration curve.*

Moisture Calibration Curve—A freshly prepared 200-lb. batch of tablets was wetted with 2000 ml. coating solution.

The tablets were allowed to tumble for 20 min. in an open pan without heat, forced air, or exhaust to achieve uniform wetting. Subsequently, three samples were taken at 15-min. intervals from different areas in the pan with the manual Analyzer electrode receptacle. Readings were taken after manipulation of the control switches on the Analyzer panel until a minimum reading on the contact meter (radio frequency conductance) was obtained. The readings in terms of units indicated the dielectric loss and, consequently, the moisture content of the tablet mass. Twenty intact tablets taken from each sample were weighed and placed in 50 ml. of anhydrous methanol, reagent grade, for 12 hr. Five milliliters of each methanol–water sample was then tested for total moisture content using the Karl Fischer method (8).

To establish a calibration curve, readings were plotted against percent moisture on a semilog graph since energy loss due to moisture is a first-order process (7). The resulting curve (Fig. 2), determined by the method of least squares, was then used to interpret readings in terms of actual tablet moisture content. This calibration curve is valid for all volumes of aqueous solution delivered. Therefore, the volumes used in the investigation were appropriate for each experiment.

Automated Coating Procedure—Two hundred pounds of freshly compressed tablets was placed in the coating pan. The automatic electrode was positioned in the tablet mass in such a manner that liquid from the spray nozzle was applied downstream in relation to tablet flow and electrode insertion. Tablet attrition on the electrode walls prevented the buildup of coating solution; therefore, electrode readings were not affected. An equilibrium meter reading of 22, corresponding to 2.2% moisture, was taken and designated as batch dryness for each experiment in the investigation. This reading was arbitrarily chosen to indicate batch dryness because it was extremely difficult to obtain lower readings within an optimum time period. The coating apparatus was put into operation by closing a master switch on the Analyzer. The contact meter indicating dryness activated the spray cycle timer (F). The timer energized a solenoid valve which, in turn, activated the automatic spray guns, allowing the flow and atomization of coating liquid. At the end of the spray cycle time, the solenoid was deenergized and the forced air and exhaust units were energized by the Analyzer. The sequence of events was repeated only when the Analyzer indicated that the predetermined degree of dryness was reached in the tablet mass. On completion of each spray cycle, the digital counter registered one coat. This coating procedure was carried out for repeated coats in each experimental portion of this investigation.

Sensitivity to Moisture Loss—Analyzer sensitivity to moisture loss was determined under four conditions: (*a*) at 29°; (*b*) at 29° with exhaust, (*c*) at 29° with forced air and exhaust; and (*d*) with forced air at 50° and exhaust.

---

[9] Boonton Polytechnic Co., Rockaway, N. J.

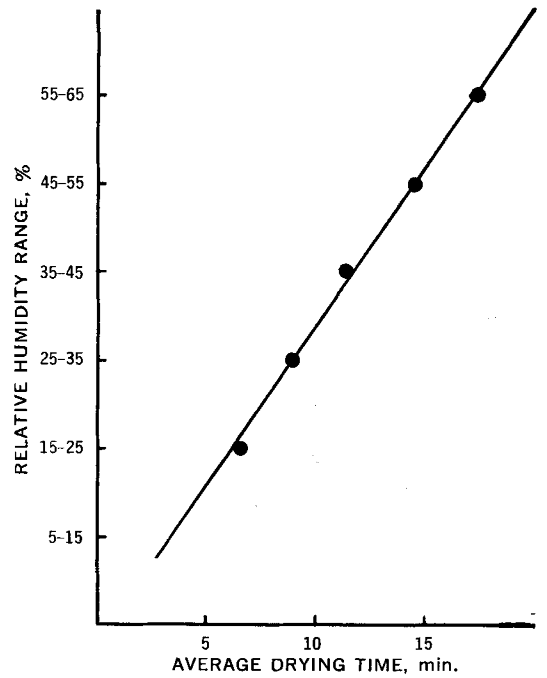

Figure 4—*Relative humidity* versus *drying time.*

The spray cycle timer was set for a 60-sec. interval to deliver 600 ml. of coating liquid and was energized manually. The wetted tablets were tumbled for 10 min. prior to manual sample testing for moisture to assure uniform distribution of coating solution. Sample readings were taken every minute after the equilibration period until a constant moisture content was attained. The coating room temperature was 29° at 60% relative humidity for the series of experiments. Each condition was carried out in duplicate with a fresh batch of tablets.

Sensitivity to Relative Humidity—The automated coating procedure was carried out on freshly prepared batches of tablets for 5 consecutive days. During this time, the ambient relative humidity varied from 15 to 60%. Coating experiments were also carried out at five controlled relative humidity range levels: 15–25 to 55–65%. Each level consisted of 10% units from a low of 15 to a high of 65% relative humidity. These relative humidity levels were attained by atomizing water into the coating-room environment. Spray cycles of 10-sec. duration, delivering 100 ml. of coating solution per cycle, were used. Moisture readings were taken every minute after an initial 3-min. equilibration period until the base reading of 22 was attained.

Sensitivity to Changes in Spray Cycle Time—These experiments were performed at a temperature of 29° and 40% relative humidity to test Analyzer sensitivity to changes in spray cycle time (change in total volume delivered). Spray cycles were begun using a 10-sec. interval and then increased by 5- or 10-sec. increments to a 30-sec. interval. Forced air at 50° was used for drying the tablet mass. Meter readings were taken every 5 min. until a plateau was reached, indicating tablet dryness.

Reproducibility of Coating Cycles—Several coating cycles were run using spray cycles of 30-sec. duration to deliver 300 ml. of coating liquid. The experiments were carried out in triplicate under conditions of 50% relative humidity at a temperature of 29°. Moisture readings were taken every minute after a 5-min. equilibration period until a plateau was reached.

RESULTS AND DISCUSSION

Sensitivity to Moisture Loss—Figure 3 shows that the Analyzer followed moisture loss with time as drying of the tablet mass occurred.[10] The differing drying curve characteristics also indicate the sensitivity of the instrument to varying drying conditions. It can be seen that forced air significantly increased the drying rate of the tablets. A plateau (batch dryness) was reached in approximately 22 min. as opposed to approximately 40 min. with and without forced air, respectively (Curves C and D *versus* A and B). Exhaust had no significant effect on the rate of moisture loss, as is shown by Curves A and B. The exhaust system could very well have been eliminated from the coating apparatus and would only be of use for collecting dust from an enclosed coating system. There was no significant difference in drying rate when comparing forced air at 29 and 50°, Curves C and D, respectively. Therefore, the major driving force for moisture loss from the surface of the wet tablets was the volume of forced air.

Table I—Effect of Relative Humidity on Drying Time

	Day				
	1	2	3	4	5
Average relative humidity, %	59.3	34.7	27.2	37.5	51.6
Average drying time, min.	18.2	10.0	9.0	11.5	15.0

Table II—Effect of Spraying Cycle Time on Drying Time

Spray Cycle Time, sec.	Drying time, min.
10	10.0
15	13.75
20	19.5
30	28.0

Sensitivity to Relative Humidity—The data in Table I show a decrease in drying time with a decrease in the relative humidity surrounding the coating apparatus. The Moisture Analyzer was quite sensitive to these changes in drying time, and a surprisingly good linear relationship was found between relative humidity and drying time when the data were plotted as a 10-unit range in percent relative humidity *versus* time (Fig. 4).

The Analyzer clearly indicated that there was a change in drying time as the relative humidity surrounding the coating system changed.

Sensitivity to Changes in Spray Cycle Time—Table II shows that the Analyzer was sensitive to a change in moisture when the spray cycle times were varied. It is evident that as the spray cycle time increased, there was a corresponding increase in drying time.

Reproducibility of Coating Cycles—The reproducibility of the coating cycle is quite evident in Fig. 5. In each case, a plateau was reached at approximately 11 min., after which the curves were linear and parallel, indicating approach to dryness.

The importance of using a moisture-monitoring device in tablet coating is illustrated in Figs. 3–5. There was an immediate indication of drying rate and drying time for the tablet mass under specific environmental and tablet mass conditions (Figs. 3 and 4). The state of the drying condition was no longer evaluated subjectively with the attendant, inherent human error; but it was evaluated continuously, quantitatively, and reproducibly as shown in Fig. 5.

In addition, the Analyzer can function as a built-in fail-safe mechanism to correct immediately the drying program of the

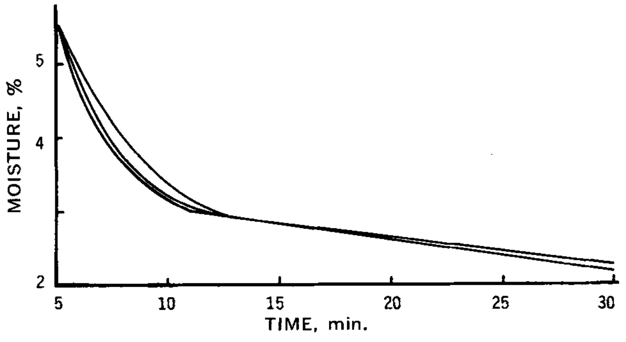

Figure 5—*Reproducibility of coating cycles.*

---

[10] Data points for Figs. 3 and 5 were omitted for the purpose of clarity.

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.

REFERENCES

(1) D. E. Wurster, *J. Amer. Pharm. Ass., Sci. Ed.*, **48**, 451(1959).

(2) R. E. Singiser and W. Lowenthal, *J. Pharm. Sci.*, **50**, 168 (1961).

(3) L. Lachman and J. Cooper, *ibid.*, **52**, 490(1963).

(4) D. S. Mody, M. W. Scott, and H. A. Lieberman, *ibid.*, **53**, 949(1964).

(5) J. Spaulding, *Drug Cosmet. Ind.*, **79**, 766(1956).

(6) G. M. Krause and T. L. Iorio, *J. Pharm. Sci.*, **57**, 1223(1968).

(7) P. S. Miller and J. J. Jones, *Proc. Sci. Sec. Toilet Goods Ass.*, No. 31, May 1959.

(8) "The United States Pharmacopeia," 16th rev., Mack Publishing Co., Easton, Pa., 1960, p. 939.

ACKNOWLEDGMENTS AND ADDRESSES

Received January 30, 1970, from the *Department of Pharmaceutics, School of Pharmacy, University of Connecticut, Storrs, CN 06268,* and *Columbia University College of Pharmaceutical Sciences, New York, NY 10023*

Accepted for publication March 23, 1970.

The experimental portions of this investigation were conducted in the research laboratories of Columbia University College of Pharmaceutical Sciences.

The authors thank Dr. Milo Gibaldi for his suggestions and helpful discussions during this investigation.

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

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.

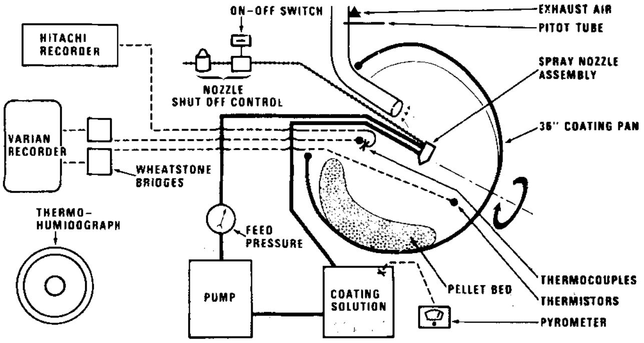

Figure 1—*Equipment diagram.*

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

---

[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.