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Material carryover and process efficiency during tablet film coating in a side-vented perforated drum (Accela-Cota)

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The application of film coatings to tablets can be carried out in a wide variety of equipment much of which has been extensively reviewed in the literature (Pickard & Rees 1974; Porter 1979). Possibly the most widely used in the pharmaceutical industry in the U.K. is the Accela-Cota (Manesty Machines Ltd, Speke, Liverpool). This comprises a perforated, cylindrical shaped drum rotating on a horizontal axis in which a charge of tablets is held and through which a current of air is continuously passed before being exhausted by means of a plenum at the back of the tablet bed. Polymer film coating solutions are sprayed on to the tablet bed by means of spray guns mounted in the drum. There is little doubt that this system is a highly efficient process, but in order to achieve some degree of control over the system as a whole, it is necessary to explore the relationships between the process efficiency with regard to solids deposition and the process variables such as spray application rate, drum rotational speed, charge size. Pickard (1979) has defined process efficiency as the ratio of the mean weight of coating found on the tablet to the mean weight of solid applied per tablet from the coating solution expressed as a percentage. The problem with this approach is that in weighing the tablets before and after coating due allowance has to be made for any loss on drying and hence the values obtained can only be regarded as approximate. We have attempted to overcome this problem by measuring the amount of solid carryover in the exhaust duct by means of isokinetic sampling, i.e. sampling at a rate such that the average air velocity entering the sampling nozzle is the same as that of the air in the duct at the sampling point. This communication reports our results and conclusions.

The sampling equipment used was the ICI standard portable gas borne dust sampling apparatus as described in detail by Stairmand (1951) and British Standard 3405 (1971). The sampling nozzle, chosen with regard to the air velocity and static head—both measured using a pitot tube–was inserted in the exhaust duct approximately 10 cm from the exhaust plenum of a 24 inch Accela-Cota. The collecting filter case containing the filter was positioned inside the Accela-Cota cabinet to avoid condensation. Material carry over was determined by weighing the filter before and after sampling. 8·5 mm diameter normal concave placebo tablets of approximate weight 200 mg were coated with a 1·5% w/v solution of hydroxy propyl methylcellulose (Shin-Etsu Chemical Co., Japan) containing glycerol (20% w/w based on polymer) and a coloured pigment (15% w/w based on polymer) dissolved in a dichloromethane–methanol (70:30% v/v) solvent system. The inlet air temperature was kept at 60 °C and any entrainment of dried spray on the internal surfaces of the drum was eliminated by correct positioning of the airless spray gun. Sampling was commenced when the system reached equilibrium and then carried out over a 15 min period. This resulted in the tablets already receiving a partial coating and avoided any dust generation from the tablets themselves during the sampling period. The coefficient of variation in the carryover results (expressed as g min^{-1}) was, in the majority of cases, better than 10%.

The results showing the effect of spray application rate (measured by loss in weight of the coating solution tank), drum rotational speed and charge size are given in Tables 1, 2 and 3. The process efficiencies are slightly higher than those previously recorded by Pickard (1979) who found values, by weighing the tablets, of between 78 and 94% for a similar solvent coating solution. Unfortunately the results cannot be directly compared because of differences in the processing conditions and film formulation.

The results indicate that a greater quantity of solid is lost and that the efficiency of the process becomes progressively lower with increasing spray application rate, increasing drum speed and decreasing charge size. The latter two relationships are thought to result from the changes in air flow through the tablet bed as a result of increased bed voidage. Pressure drop measurements across the tablet bed which showed a progressive decrease with these two variables support this. Qualitative measurements using a muslin filter wrapped around the drum showed that most of the carryover was concentrated around the baffles, indicating that it is through these regions of low tablet bed density that most air passes.

Table 1. The effect of spray rate on material carryover and process efficiency (drum speed 16 rev min^{-1}, charge 10 kg).

Approximate spray rate* cm^3 min^{-1}	200	255	290	315
Solids application rate† g min^{-1}	5·30	7·29	8·45	8·53
Material carryover g min^{-1}	0·039	0·136	0·302	0·354
Material carryover %	0·74	1·86	3·57	4·15
Process efficiency %	99·3	98·1	96·4	95·8

* Extrapolated from pump setting.
† Measured by weight difference.

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Table 2. The effect of drum rotational speed on material carryover and process efficiency (solids application rate 7·30 g min^{-1}, charge 10 kg).

Drum rotation speed rev min^{-1}	12	15	20	24
Material carryover %	1·81	1·85	2·13	2·21
Process efficiency %	98·2	98·1	97·9	97·8

Table 3. The effect of charge size on material carryover and process efficiency (solids application rate 7·30 g min^{-1}, drum speed 16 rev min^{-1}).

Charge size kg	6	10	12
Material carryover %	3·25	1·85	1·82
Process efficiency %	96·7	98·1	98·2

These results have important practical implications in the film coating of solid dosage forms and are particularly relevant to sustained release products where the drug release rate is dependent on, inter alia, the thickness and the uniformity of the film coating. In this case increasing the drum rotational speed may well be detrimental since, although improving mixing of the tablet bed, it may well result in increased carryover and decreased film thickness. The results illustrate the potential of this accurate, rapid and simple technique of isokinetic sampling of the exhaust duct in the optimization of process conditions during product development.

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REFERENCES

British Standard 3405 (1971) British Standards Institute, London

Pickard, J. F. (1979) PhD thesis. Council for National Academic Awards

Pickard, J. F., Rees, J. E. (1974) Manufacturing Chemist Aerosol News 45: 42–45

Porter, S. C. (1979) Pharm. Tech. 3: 55–59

Stairmand, C. J. (1951) Trans. Inst. Chem. Engrs. 29: 15–44

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Determination of salicylic acid in acetylsalicylic acid by second derivative u.v.-spectrophotometry

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Salicylic acid (SA) is the major decomposition product of acetylsalicylic acid (ASA). It is well known that the free acid can cause gastric diseases; therefore several methods have been developed for its determination in ASA pharmaceutical preparations.

These methods have employed partition chromatographic (Levine & Weber 1968; Guttman & Salomon 1969), gas liquid chromatography (Galante et al 1981), liquid chromatography (Baum & Cantwell 1978), high power liquid chromatography (Ali 1976; Das Gupta 1980; Kirchhoefer 1980; Kirchhoefer & Juhl 1980), spectrophotometric (Reed & Davis 1965; Clayton & Thiers 1966), colorimetric (Kubin & Gaenshirt 1976; Juhl & Kirchhoefer 1980) and fluorometric (Shane & Miele 1970; Shane & Stillman 1971; Schenk et al 1972) procedures.

This paper reports a simple, rapid and accurate method for the direct determination of salicylic acid in ASA by second derivative u.v.-spectrophotometry. Fig. 1 shows the zero order and second derivative ultraviolet spectra of solutions of ASA, SA and ASA with SA in dioxane, and the method of measurement.

Materials and methods

Apparatus. The spectra were obtained with a Perkin-Elmer Model 200 ultraviolet-visible spectrophotometer, equipped with a Hitachi electronic derivative module. Derivative conditions: scan speed 240 nm min^{-1}; spectral slit width 2 nm; mode (time constant) No. 6; response slow.

Reagents. Salicylic acid was analytical reagent grade. Acetylsalicylic acid used for the control analytical curves was prepared by purifying the pure commercial product with ten crystallizations from anhydrous methylene chloride. Dioxane was spectroscopic reagent grade. Dry ethyl ether used for the extraction of the pharmaceutical forms was prepared by a Brockmann zero basic alumina column.

Pharmaceutical preparations analysis. The powdered pharmaceutical form was rapidly extracted with anhydrous ethyl ether at room temperature. The ether solution obtained after filtration was evaporated under reduced pressure and the residue rapidly dissolved in dioxane. The concentration of this last solution was related to the amount of SA in the sample. The second derivative of the u.v.-spectrum was registered and the peak-trough amplitude at 308 nm was

Table 1. ASA concentrations in the sample solution related to the SA concentration in the sample.

SA (ppm)	ASA (mg ml^{-1})
10–100	15
100–1000	1·5
1000–50 000	0·15

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