Adaptation of Commercial Viscometers for Special Applications in Pharmaceutical Rheology III. The Tackmeter

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A variant of the tackmeter originally developed by Green has been constructed. This instrument is suited to the precise measurement of tack instead of the usual subjective evaluation of lotions, creams, and ointments. It may be used in laboratory screening to compare relative tack of different formulations as a function of drying time or conditions and as a control procedure for formulations. It has been usefully applied as a measure of the tack left by materials rubbed into the human skin.

IN THE APPLICATION of lotions, ointments, or creams to the skin for therapeutical and/or cosmetic purposes, the resultant tackiness or stickiness of the area is often of concern—because of skin to skin adhesion for certain areas of the body or because of adhesion to a protective dressing.

The evaluation of the relative merits of different formulations has primarily been subjective. It was felt desirable to quantitate this, preferably by a direct measure which could compare similar formulations and also determine any aging changes as they might occur.

The problem of tack has been a continuous one in the printing industry. Green published a description of a tackmeter (1) which was, "a mechanical finger, paralleling in execution the finger tap-out test in the evaluation of pull-resistance, but capable of placing a numerical value on its measurements."

The instrument described here is a simplification of the Green tackmeter which permits evaluation of tack of lotions, creams, and ointments, both on air drying on plates and after application—with or without rub-in on the skin.

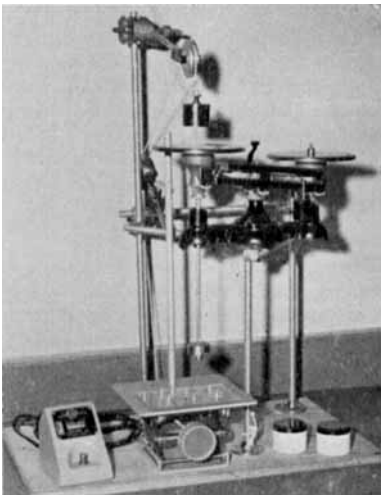

Fig. 1.—The tackmeter assembly with outer cup rotated upward to display the finger.

EXPERIMENTAL

The tackmeter assembly is shown in Fig. 1. The balance is a double beam Ohaus, Harvard Trip model. The assembly is connected to the beam collar underneath the frame casting by a connector and pin. The pin and hole in the vertical shaft is already present in this model. The collar must leave sufficient clearance for the bottom arm of the parallelogram of the balance to move freely. The connecting rod ($^1/_4$ in.) may be of any convenient length. The rod is connected to the collar by a set screen to permit easy removal for cleaning. The finger assembly is as shown. The finger diameter is $^5/_8$ in.; the inner diameter of the cup is $1^1/_4$ in. The thread for the rotation of the cup is 28 per inch. Two $^3/_4$-in. slots were cut in the cup to permit viewing the finger in operation. The whole assembly was of 316 stainless steel construction.

In use, the procedure may vary somewhat with the type of material being studied. For the following of the tack on air drying of a lotion, rings of 25-mm. glass tubing in $^1/_4$-in. lengths were cemented with Eastman 910 adhesive to a glass plate. A measured volume, 0.2 ml., was transferred to this cup and allowed to dry under prescribed suitable conditions. The finger was recessed to give the desired thickness of test lotion. To do this, the finger was contacted by a planar surface; then the cup was rotated downward until it, too, was in contact. Since the outer wall of the cup was scribed at every 10°, angular rotation of the cup could be readily determined using the pointer attached to the rod (and visible in the figure). The cup was rotated until the finger was recessed to the desired distance. Each 10° of rotation represented was 0.000992 in. of relative motion. The balance was counterweighted for the weight of the tackmeter. A 500-Gm. weight was placed on the left-hand pan and the plate carrying the dried lotions raised sufficiently by a jack, so that the finger could be located in the center of the cup. The jack was then further raised so that the cup bore on the plate. The desired drawing weight was placed on the right-hand pan. A stop clock was started as the 500-Gm. weight was removed; the time for the finger to break clear was noted. For the comparison of notably different lotions, the weight required to break clear in 6–10 seconds was determined, while for very similar lotions the differences in time for the same load were determined. Typical data are reported in Table I.

For ointments and creams, several modifications of this procedure were satisfactory. A film could be drawn by a doctor plate technique if the material

TABLE I.—COMPARISON OF TACKINESS OF SEVERAL LOTIONS ON DRYING

Sample	Withdrawal Wt. and Time of Break — 3 Hr. Drying	6 Hr. Drying
1	2 Gm. for 65 sec.	20 Gm. for 30 sec.
2	2 Gm. for 1.1 sec.	2 Gm. for 6 sec.
3	2 Gm. for 10 sec.	15 Gm. for 15 sec.
4	2 Gm. for 2.7 sec.	3 Gm. for 19 sec.

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TABLE II.—COMPARISON OF TACKINESS OF SEVERAL CREAM PREPARATIONS RUBBED IN THE SKIN FOR 20 SECONDS

Sample	Withdrawal Wt., Gm.	Time for Withdrawal, sec.
1	4	129, 165, 234, 212
2	4	101, 65, 71
3	2	6, 2, 2

was not too shear sensitive. Alternately, a weighed geometric spot was applied to the glass. In either case, the finger was brought to bear in a similar manner.

For actual measurement on human skin, the sample was applied in the appropriate manner and the finger applied directly with the 500-Gm. load. The cup was rotated back and clear for this. In general, the finger was contacted for 20 seconds before withdrawal; voluntary motion or quivers often induce the break-away after longer periods of contact. Typical data are shown in Table II.

DISCUSSION

The fundamental equations covering the use of the tackmeter were derived by Green (1), and the rheology of tack has been discussed by Bikerman (2).

The time required for break is inversely proportional to the viscosity function of the sample, the weight used for drawing, and the square of the sample film thickness, but is proportional to the fourth power of the radius of the finger. Thus, it is possible to derive a viscosity term from the tackmeter to define the sample film. In this work, it has not been necessary, or even desirable, to do this, since all desired comparisons can be readily made in units of withdrawal time and pulling weight. If, however, some knowledge of absolute viscosities of dried films is desirable, then the instrument may be calibrated by standards and used as a rheometer.

Obviously, the choice of finger dimensions and film thickness permit an extremely wide range of study. In the study on human skin, the bearing weight and duration of contact could conceivably be of different magnitudes in some applications.

Since the primary value of this instrument is for empirical testing, the finger surface may readily be modified to permit the attachment of dressings if it is felt to be important.

SUMMARY

The tackmeter described permits the measure of tack of lotions, creams, and ointments. It may be utilized on human skin and as a laboratory test.

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Assay Methods for Total Neomycins B and C

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Data from assays of neomycin C sulfate and mixtures of neomycin C sulfate and neomycin B sulfate were obtained using six cylinder-plate methods. Statistical analyses show that with three commonly used methods, neomycin C responses are only 35 to 50 per cent of neomycin B responses. With three modified methods, the response of neomycins B and C are approximately equal.

NEOMYCIN FERMENTATIONS produce two major antibacterial components—neomycin B and neomycin C. Commercial preparations are composed mostly of B, but may contain as much as 50% C (1). Kaiser (2) found that the U.S.P. neomycin standard contained about 15% C. The present assay systems do not give equivalent responses for B and C and with significant differences in C content between the test and standard preparations, the assay results are not in terms of total neomycin relative to the standard.

Some problems in the assay of neomycin were discussed in a previous paper (3). The purpose of this study was to compare responses of neomycin B and neomycin C obtained by three assay methods commonly used and by three methods which were designed to and did reduce differences between the responses of neomycins B and C.

EXPERIMENTAL

Neomycin Preparations.—The physical and chemical properties of the neomycin B preparation and the neomycin C preparation were described by Ford, *et al.* (4). With a radioisotopic assay procedure (2), the neomycin B preparation was found to contain 11% C, while the neomycin C preparation was found to be free (<1%) of neomycin B. Both preparations were sulfate salts, and potency values for Tables I–III were recorded as neomycin sulfate. The potency values for Table IV are recorded in terms of neomycin base equivalent.

The Upjohn Co. control standard, potency 742 mcg./mg., was used to determine the potencies of commercial neomycin sulfate lots (Table IV).

Preparation of Neomycin Solutions.—The neomycin powders were weighed after drying in a vacuum oven at 60° for 3 hours. One milligram per milliliter stock solutions were made with distilled water. Stock mixtures in ratios of 1:1 and 9:1 (9 neomycin B + 1 neomycin C) were prepared from these solutions. Working solutions were prepared by diluting the stock solutions with the buffer appropriate to the method.

Assay Methodology.—The cylinder-plate method was used for all assays. Twenty by 100-mm. plastic Petri dishes (Plastomatic Corp., Malvern, Pa., cat. No. 94), were used with glazed porcelain tops. Six stainless steel cylinders (inside dimensions 6 × 10 mm.), were placed on each plate with a Shaw

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