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Quality control of white soft paraffin

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Samples of white soft paraffin complying with pharmacopoeial requirements vary in their physical properties. Some samples produce hard waxy lumps and permit excessive separation of liquid components (bleeding). Measurement of melting and congealing points and consistency by penetrometer failed to reveal these differences. The viscosity of samples after 20 min shear ($1159 s^{-1}$) is related to lump formation as detected by usual examination of samples heated and cooled without stirring and spread on a tile after gentle mixing.

Various tests have been applied in the United States and British Pharmacopoeial monographs in attempts to control the physical properties of white soft paraffin. It is our experience that these tests are inadequate to determine the tendency of samples to form a hard waxy crust when heated and cooled without agitation, and the dispersion of the crust as hard waxy lumps on stirring the product, and also the separation of liquid components (bleeding) particularly after shearing and storage at elevated temperatures.

The chemical composition and difficulties involved in the definition of white soft paraffin have been described by Schulte & Kassem (1963) and by Franks (1964). The presence of n-, iso- and isocyclic paraffins commonly referred to as paraffin, intermediate and microcrystalline waxes respectively is recognized. The liquid paraffin fraction is believed to be held in a gel structure within a network of microcrystalline waxes. The rate of structural breakdown after shearing and the time for regeneration of structure have been related to the composition of the white soft paraffin by Schulte & Kassem (1963). Van Ooteghem (1968) has suggested that the quality of white soft paraffin may be determined by measurement of the speed of breakdown of its structure under shear at a rate of shear of approximately $250 s^{-1}$. Boylan (1966, 1967) has investigated the thixotropic behaviour of white soft paraffin and has suggested that comparison of the hysteresis loop of samples and a standard will provide a measure of the spreadability of the product.

Whilst the definition of "quality" in white soft paraffin varies with individual formulators' ideas and the intended use of the end product, it is clearly desirable that lump formation and bleeding should be minimized. The work in this paper describes our attempts to initiate test procedures designed to select those paraffins free from such undesirable tendencies and to illustrate the inadequacy of the present pharmacopoeial standards.

EXPERIMENTAL

Samples of white soft paraffin (A–P, Table 1), claimed to be of British Pharmacopoeial quality, were obtained from several manufacturers. Samples 1–5 (Table 2) of known composition were obtained from one manufacturer. All were subjected to the tests described below:

(1) *Tendency to form lumps.* Each sample (20 g) of white soft paraffin was heated to 80° in a 50 ml beaker, cooled to 25° over 2 h, mixed by stirring gently with a glass rod, spread on a black tile and observed for the presence of hard waxy lumps.

(2) *Bleeding tendency.* Samples (30 g) were treated as described in (1) above up to the mixing stage and then subjected to a test based on that of Van der Pol (1960). Strips of Whatmans No. 1 filter paper (2 × 25 cm), supported vertically above the beaker, were inserted in the unmixed samples to a depth of 2 cm and the height of ascent of oil after 24 h at 25° was measured. The height of ascent expressed in mm is the bleeding number.

(3) Melting and congealing points were determined as described in the British and United States pharmacopoeias respectively.

(4) *Unctuous nature.* The samples pretreated as in (1) above were assessed subjectively as described in the British Pharmacopoeia.

(5) *Consistency.* Penetration values were determined using a Stanhope penetrometer in combination with a cone shaped metal plunger weighing 7·5 g. Samples were prepared for testing by the U.S.P. method to produce an unworked sample. Certain samples were subjected to standard shear by repeated forcing through a narrow aperture (standard working conditions) and their "worked penetration" value determined immediately. The results are expressed as the average of three determinations.

(6) *Viscosity.* Samples pretreated as described under (2) above were introduced to the plate of the Rotovisko viscometer PK1 system with minimal shear. The samples were sheared at speed 9 (rate of shear 1159 s^{-1}) or speed 54 (193 s^{-1}) and the fall in viscosity with time and the final viscosity after 20 min shear were recorded. The pretreatment of samples and minimal shear involved in the introduction of samples to the cone and plate system ensured greater reproducibility of results than could be obtained using other systems.

RESULTS AND DISCUSSION

The results presented in Tables 1 and 2 show that all samples tested comply with the B.P. and U.S.P. tests for melting and congealing points. Although samples A–P can be described as unctuous to the touch, their tendency to lump formation and bleeding vary considerably.

Consistency determinations by penetrometer methods are widely used for quality control of white soft paraffin. The U.S.P. method for the preparation of samples for such determinations involves heating and cooling of samples without stirring, to ensure homogeneity. This may result in the formation of a waxy surface which in samples 1, 2 and E, F, G, K led to low penetration values suggestive of a paraffin of high consistency. Subjection of samples 1 and 2 to standard working conditions showed the structure to be rapidly destroyed, the resulting samples being too fluid for consistency determinations. Comparison of the penetrometer values and final

Table 1. *Melting and congealing points, penetrometer values, viscosity and tendency to form lumps of samples of white soft paraffin*

Sample	M.p. °C	Congealing point °C	Penetrometer (mm)	Viscosity cP P.K.1 system shear rate 1159 s^{-1}	Lumpiness
A	50	50	39·2	1390	—
B	50	51	43·6	1495	—
C	52	51	20·3	3065	—
D	52	53	27·0	970	—
E	47	51	30·5	334	+
F	51	50	17·7	160	+
G	50	51	22·3	300	+
H	56	56	24·5	760	—
I	51	55	36·3	960	—
J	51	52	29·8	1250	—
K	57	59	33·3	180	+
L	47	51	38·7	600	—
M	52	55	39·3	780	—
N	51	53	30·3	750	—
O	47	51	37·0	499	—
P	51	56	32·6	1320	—

Table 2. *The physical properties of samples of white soft paraffin of known composition*

	Sample 1	2	3	4	5
Composition % by weight					
Paraffin wax	15	—	—	—	—
Intermediate wax	—	14	10	7	—
Microcrystalline wax	—	—	10	28	50
White oil	85	86	80	65	50
Test					
Lump formation	+	+	—	—	—
Bleeding No.	41	32	30	10	4
Consistency (mm) unworked	12·3	26·6	49·9	28·8	16·7
(Penetrometer) worked	Fluid	Fluid	75·2	43·2	31·8
Melting point °C	38	44	49	51·5	53
Congealing point °C	42	50	52	53	51
Viscosity (cP) (1159 s^{-1})	194	281	359	1748	2893

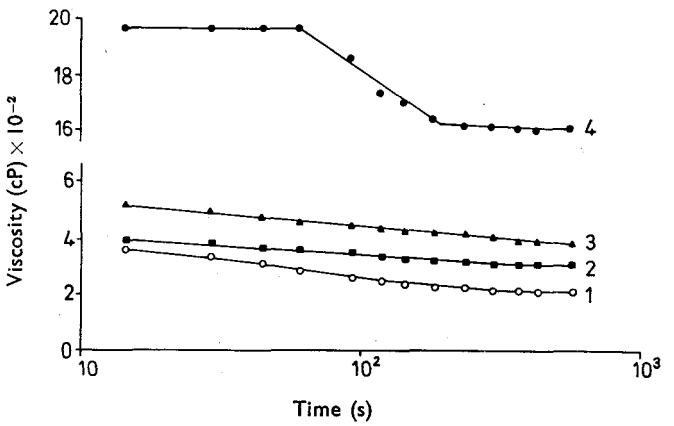

FIG. 1. Effect of time of shear (shear rate 1159 s^{-1}) on the viscosity of samples 1–4.

viscosities recorded in Tables 1 and 2 indicates that the penetrometer does not give a true measure of consistency when testing samples which tend to form lumps, nor do the results reflect the differences in viscosity between samples as measured by the Rotovisko.

The change in viscosity of samples 1–4 with time when sheared at a rate of 1159 s^{-1} are shown in Fig. 1. Measurements made at a shear rate of 193 s^{-1} produced results comparable with those shown. However, much difficulty was encountered when using this lower shear rate with samples of high viscosity and the results were not reproducible. The effect of time of shear (1159 s^{-1}) on the structural breakdown of samples A–P was similar to that of samples 1–3 shown in Fig. 1. In all cases structural breakdown was complete within 20 min of the application of shear. The final viscosity of the samples after 20 min shear are shown in Tables 1 and 2. One sample tested showed unusual behaviour and reproducible results were difficult to obtain. This sample tended to be thrown out from between the cone and plate of the viscometer.

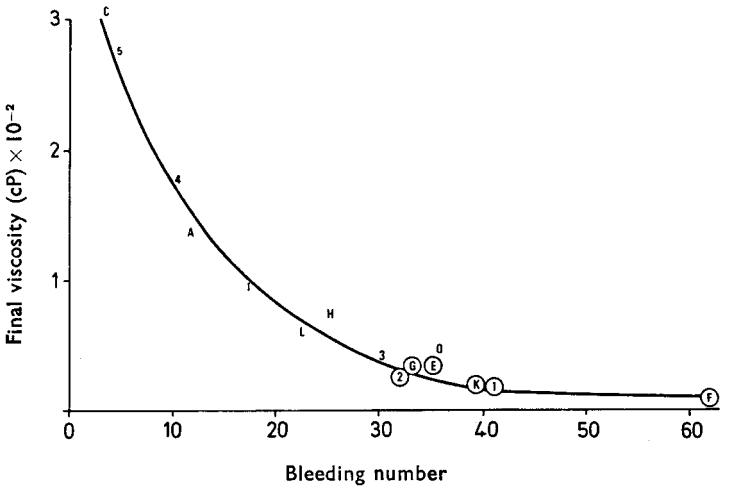

FIG. 2. The relation between final viscosity and bleeding number of samples of white soft paraffin. Those ringed samples produced lumps.

The relation between final viscosity, bleeding number and tendency to form lumps is shown in Fig. 2. The tendency of the liquid fraction of white soft paraffin to separate and for lumps to form is greater with samples of lower viscosity. Sample O, of relatively low viscosity, is an exception in that it does not form lumps.

Difficulties involved in the chemical analysis of white soft paraffin are such that it is unlikely that chemical definition of the product is practical. Attempts to use gas-liquid chromatography to determine differences in the composition of samples revealed that such differences existed but it was not possible to relate results to the physical properties of the samples. Franks (1964) believes that a strong stable homogeneous gel structure capable of withstanding repeated shear is perhaps the most important characteristic of white soft paraffin. The results in Table 2 and Fig. 3 show that an increase in microcrystalline wax content and therefore in network structure produces an increase in final viscosity with a reduction in bleeding tendency.

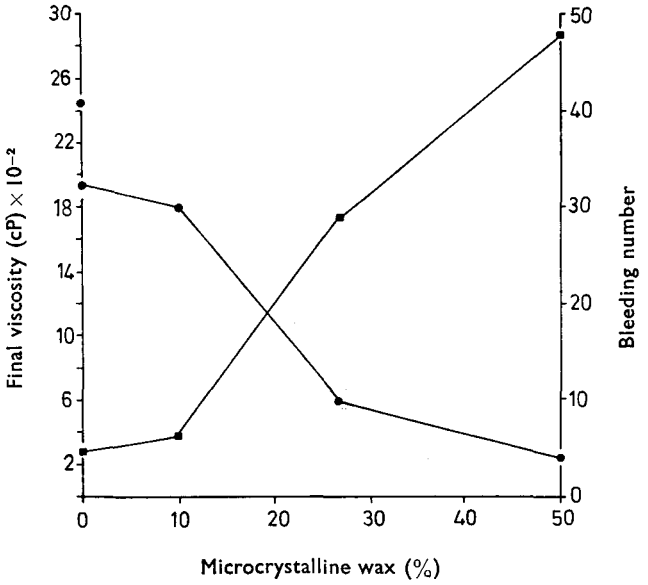

FIG. 3. The effect of microcrystalline wax content on the final viscosity and bleeding number of samples 2–5 of white soft paraffin. ■—■ Final viscosity. ●—● Bleeding number.

In our experience two extra tests must be added to those described in the B.P. These are the measurement of the final viscosity after shearing and the tendency of the sample to form lumps. With these, adequate information is available to predict the acceptability of a particular sample for use in ointment formulations. The use of rate of structural breakdown as a measure of performance is unnecessary and difficult to determine. At present the finer qualities of white soft paraffin such as fibre length and spreadability are best measured by subjective tests, but the application of the tests for lumpiness and viscosity described can minimize problems of lump formation and bleeding.

REFERENCES

BOYLAN, J. C. (1966). *J. pharm. Sci.*, **55**, 710–715.
BOYLAN, J. C. (1967). *Ibid.*, **56**, 1164–1169.
FRANKS, A. J. (1964). *Soap Perfum. Cos.*, **37**, 221–230; 319–326.
SCHULTE, K. E. & KASSEM, M. A. (1963). *Pharm. Acta Helv.*, **38**, 358–370.
VAN DER POL, H. J. (1960). *Pharm. Weekblad*, **95**, 3–7.
VAN OOTEGHEM, M. (1968). *Pharm. Acta Helv.*, **43**, 264–272.