

ON A NEW FORM OF REAGENT AGAINST PERSPIRATION EFFECTS ON SHOE MATERIALS

By A. COLIN-RUSS, Ph.D., F.R.I.C., *From the Research Association Laboratories of
the British Boot, Shoe and Allied Trades Research Association*

CONTENTS

	PAGE
Introduction	53
Experiments	53
(1) General procedure	53
(2) Activity of emulsifying components	54
(3) Influence of lactic-urea-salt ingredients	54
Standard perspiration reagent (Conc.)	54
References	55

INTRODUCTION

In a discussion of a series of tests connected with the fastness of colours on textile fibres, Villavecchia (1918) mentions the use of neutral ammonium acetate or common salt solution for measuring the colour resistance to perspiration. Thus in the case of coloured cotton, this is immersed for 10 min. alongside an equal quantity of white cotton yarn in a 0.1 % aqueous ammonium acetate at 80°C., and the extent of colouring of the yarn as well as the degree of stripping of the coloured sample noted after drying without rinsing. An odd number of degrees of fastness is arbitrarily assigned to the coloured specimens according to the result, e.g. V degrees if neither the original tint nor the whiteness of the yarn is changed. Similarly with linen, hemp and ramie. For coloured wool both methods are used, viz. with sodium chloride and with ammonium acetate. In the former case, the alteration in colour is noted after simply dipping the wool in salt solution and allowing to air-dry out at room temperature. In the latter case, an equal quantity of white zephyr wool in addition to the white cotton is present. V degrees in all cases are stated to be conventionally adopted between industrial associations and dyestuff manufacturers for materials which are fast enough to reveal no change whatever in the testing bath. For coloured silk there are no precise data, and usually it is immersed in distilled water for several days to ascertain if any colour is lost.

From a scientific point of view, ammonium acetate or common salt represents only a crude approximation to the composition of perspiration itself. The restriction of ammonium acetate to the neutral state is itself partially nullified, and fortunately so by the hydrolysis on warming, since perspiration has a wide pH range of activity.

There may have been in the past very close cor-

relation between the tests and the actual wear of the material against perspiration, but it must be admitted that only on the sure foundation of an effective composition for perspiration can the latter's influence be truly gauged.

Scattered in the literature, the above form of tests with minor variations is typical, and for the case of coloured leather there is no obvious advantage in dipping into salt solution instead of water, although warm immersion for a period would be. Furthermore, colouring matter is not the only component of a material which is required to be fast to perspiration, and this statement applies *a fortiori* to leather whatever its tannage.

In a series of studies by Colin-Russ (1935, 1940, 1943) on foot perspiration and its effects on footwear, the consideration of perspiratory constituents individually led to interesting observations on the changes in composition which leather material can undergo.

With the experience gained in the use of a synthetic type of perspiration as reagent and the need to carry out a large number of routine analyses on the material tested under a wide variety of conditions, it was natural to seek maximum simplification of reagent composition consistent with the true character of the results. Fortunately, such simplification led to the added convenience of pleasant operation coupled with non-fouling of the laboratory atmosphere.

The following experiments form the basis of the simplified formula finally adopted.

EXPERIMENTS

(1) *General procedure*

Of the nine components given in the formula for synthetic preparation by Colin-Russ (1935, p. 202), three only were chosen for possible elimination, viz.

butyric acid, cholesterol and phospholipin. The tallow was deemed too weak for its small potential contribution of fatty acid to be essential, and the phosphate solution, though equally small in amount, was retained as a buffer.

Each of the three chosen components was tested in the concentration existing within the 10 % strength of synthetic perspiration against a chrome leather sample of known loss of sulphate when treated with full synthetic preparation as prepared free from its normal potassium sulphate component.

The test was next repeated with the full synthetic preparation deprived of the butyric acid component in addition to its normal sulphate, although the loss of sulphate from the leather due to the butyric constituent was appreciable. As the subsequent full loss of sulphate in the test was obtained equally as well as with the butyric acid present, the test was again repeated with the omission altogether of tallow, phospholipin and cholesterol in addition to butyric acid and potassium sulphate. This proved to be the simplified form, since the liberated ionizable sulphate from the leather material remained substantially the same with the reagent hitherto used in past work.

(2) Activity of emulsifying components

Experiment 1

A. *With butyric acid alone.* 5 ml. of butyric acid were delivered from a pipette into a mixture of 140 ml. water and 5 ml. alcohol. From this solution, an aliquot of 10 ml. was withdrawn into a 100 ml. graduated flask and finally made up to the mark with water. This reagent containing $\frac{1}{3}$ % w/v of butyric acid equivalent to the 10 % synthetic perspiration of previous work, was boiled under reflux for 1 hr. with 5 g. box calf '613', and then the sulphate was determined.

B. *With saturated alcoholic egg lecithin.* 5 ml. of this alcoholic solution were further diluted with alcohol to 70 ml. and then 10 ml. withdrawn into a 100 ml. graduated flask for making up to the mark with water. The subsequent procedure was the same as A.

C. *With cholesterol alone.* 1.43 g. of 'Hartolan' was dissolved in 10 ml. warm alcohol, then made up to the mark with distilled water in a 100 ml. graduated flask. The reflux procedure of A or B was applied in this case, too.

The following results were obtained:

	% SO ₄ lost on original
A	15.55
B	14.75
C	16.00

Previous work by Colin-Russ (1940, Table 1, p. 450) indicated a loss of 54.6 % with the same

leather sample. On another occasion (Colin-Russ, 1943, p. 74) it was 57.5 %. Thus, although approximately a third only was yielded in this experiment, the loss cannot be ignored, and the evidence so far does not yet justify the elimination of the components in A, B or C.

(3) Influence of lactic-urea-salt ingredients

Experiment 2

This was divided into three parts; one part with boxcalf '613' and the other two with boxcalf '4470'.

(I) 10 % aqueous synthetic perspiration excluding the potassium sulphate and butyric components was boiled for 1 hr. under reflux with leather '613' in the proportion of 100 ml. to 5 g. leather. Liberated =SO₄ was 58.0 % as based on the original total.

Thus although butyric acid alone can yield almost one-third of this value, its absence had no effect on the superior decomposing power of the remaining ingredients, which incidentally was evident in a similar chrome oxide loss, viz. 3.78 % compared with 3.4 % (Colin-Russ, 1940).

(II) In order to confirm the higher activity of these ingredients, the foregoing was repeated with boxcalf '4470' which was known hitherto to lose 33.5 % of its total sulphate. The result obtained of 33.3 % thus agreed closely.

(III) In order to set this confirmation beyond doubt and simultaneously to test the most simplified form of perspiration reagent, (II) was repeated in the absence of 'Hartolan', egg lecithin, tallow, as well as butyric acid and potassium sulphate. The yield of =SO₄ proved to be 33.9 %.

The following formula is therefore given with the assurance that it has been found serviceable and pleasant in the course of doing literally hundreds of routine treatments of fabric and leather materials.

Standard perspiration reagent

(Conc.)

Urea crystals	1 g.
Lactic acid B.P.	5 ml.
10 % aq. disodium phosphate	1 ml.
N sodium chloride solution	100 ml.
Distilled water added	500 ml.

For use equivalent to 10 % synthetic perspiration solution of the old emulsion form, 35.55 ml. of the above solution are measured from a burette for subsequent dilution to 100 ml. Since, in practice, an aliquot of the dilution is required for initial pH adjustment, it has been a routine procedure to withdraw 37.35 ml. for dilution to 105 ml. Thus, apart from an ample margin of 5 ml. for pH measurement, there is freedom to admix other

solutions such as tannin, buffer, etc., before attainment to the 105 ml. graduation.

The formula of the standard perspiration reagent is offered as being comparatively simple in character, is obviously susceptible of easy analytical control and could be modified into compressed tablet form if desired. Furthermore, should a particular investigation require the use of synthetic perspiration as an emulsion to represent sweat and sebum, this is easily obtained by gradually adding

0.5 l. of the standard solution at 35° C. to a molten mixture of

Hartolan	20 g.
Butyric acid	5 ml.
Saturated alcoholic egg lecithin	10 ml.
Russian tallow	1 ml.

and using a powerful whisk.

The author's best thanks are due to the Director and Council for permission to publish.

REFERENCES

- COLIN-RUSS, A. (1935). *J. Hyg., Camb.*, **35**, 199–206.
 COLIN-RUSS, A. (1940). *J. Hyg., Camb.*, **40**, 447–452.
 COLIN-RUSS, A. (1943). *J. Hyg., Camb.*, **43**, 72–82.
 VILLAVECCHIA, V. (1918). *Treatise on Applied Analytical Chemistry*, **2**, 514–17.

(MS. received for publication 23. XII. 43.—Ed.)