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**Autoclave Setting of Wool Yarn**

**Part I: A Study of the Effect of Setting on  
the Rates of Exhaustion of Certain Dyes**

**by**

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## AUTOCLAVE SETTING OF WOOL YARN

### PART I : A STUDY OF THE EFFECT OF SETTING ON THE RATES OF EXHAUSTION OF CERTAIN DYES

by N. J. J. VAN RENSBURG

#### ABSTRACT

*The effect of autoclave setting of wool yarn on the rates of exhaustion of different types of wool dyes; namely the 1:1 metal complex, 1:2 metal complex, acid milling, acid levelling and reactive dyes, was investigated. Setting at high temperatures (110°C and 120°C) increased the rates of exhaustion of practically all the dyes. Some dyes were affected to a greater extent than other dyes.*

*The effect of setting on the degree of whiteness, urea-bisulphite solubility, breaking strength and snarling twist of the yarn was also investigated.*

#### KEY WORDS

Wool yarn — autoclave setting — snarling twist — dyeing — rate of exhaustion — degree of whiteness — urea-bisulphite solubility.

#### INTRODUCTION

The liveliness of a newly spun wool yarn can cause problems during weaving or knitting. The liveliness of the yarn gradually decreases upon storage under ambient atmospheric conditions. This, however, is a very slow process. Results published by WIRA show that the snarling twist of a wool yarn decreased by 30 per cent after storing for three days, and by 56 per cent after eight months<sup>(1)</sup>. It is not always practical to store the yarn for such an extended period, and the yarn is therefore subjected to a relatively short setting treatment after spinning to prevent the formation of snarls and kinks. Setting is normally achieved by steaming the yarn in an autoclave. The wet durability of the set of the yarn twist is not important, the main objective of the treatment being the elimination of processing difficulties caused by the liveliness of the newly spun yarn.

Steaming, however, can affect certain properties of the wool fibre. One of the most important modifications caused by steaming or setting of the wool, is a change in the dye affinity of the fibre. When yarns set under different conditions are mixed, or when an uneven setting of yarn in a lot occurs, there may be differences in the dye affinity of the different yarns, which may result in unlevel dyeings. This phenomenon is said to be encountered quite frequently in industry, and yet relatively few publications about the effect of setting on the dyeing behaviour of wool have appeared. Furthermore, Henning and Sustman<sup>(2, 3)</sup> pointed

out that the results published by the various authors were not always in agreement. Some of these anomalies, however, might have been due to the fact that different dyes and different types of wool which might have reacted differently to the setting treatments, were used by the various research workers. O'Reilly and co-workers<sup>(4)</sup>, for example, used Anthraquinone Blue SKY (C.I. Acid Blue 78) and found that the rate of dye-uptake by the wool was reduced by a steaming treatment. Henning and Sustman used Crystal Ponceau 6R extra (C.I. Acid Red 44) and found that the dye-uptake of the wool increased after steaming in the wet state, and decreased after steaming in the air-dry state<sup>(2)</sup>. Another publication<sup>(5)</sup> showed that the affinity of an acid dye for wool increased with increased steaming times. It was not specified, however, which dye was used for the investigation. Leidelmeyer and Pleumeekers<sup>(6)</sup> found that the dye-uptake of New Zealand and South American wool increased significantly when steamed wet, while Australian wool showed little difference. The observed differences were ascribed to differences in the pH values of the aqueous extracts of the undyed yarns. The authors found that the dye-uptake increased as the time or temperature of steaming was increased. Robinet and co-workers<sup>(7)</sup> found that steaming of wool did not affect the uptake of Carmine Blue V (C.I. Acid Blue 3), while Alizarine Brilliant Sky Blue R (C.I. Acid Blue 62) was affected very slightly. They found that the yellowing of the wool due to steaming was generally much more pronounced than were the observed differences in dye-uptake. Lagermalm and co-workers<sup>(8)</sup> found that the uptake of Naphtalene Scarlet 4R 120 (C.I. Acid Red 18) by wool was not influenced by steaming. Mihalik and Asboth<sup>(9)</sup>, on the other hand, found large differences in dye-uptake between yarn from the inside and outside of a spinning cop. They did, however, use extreme setting conditions, namely 130°C for 15 to 60 minutes.

Obviously there is a lack of information on this subject and a detailed investigation into the effect of setting conditions on the affinity of wool for various dyes will probably be of importance to spinners who are setting the yarn, as well as to dyers who have to dye these yarns subsequently.

## EXPERIMENTAL

A commercial wool yarn, spun at a South African textile mill, was used in the study of the effect of setting on the rates of exhaustion of certain dyes. The characteristics of the yarn, which was *not steamed after spinning*, are given in Table I. An aqueous extract of the yarn had a pH of 9.3. The yarns were conditioned at 65% RH and 20°C for 24 hours before setting. Approximately 40 g of the yarn was steamed on spinning cops under various conditions in an Andrews Auto-setter (cubic capacity 2 662 litres). The autoclave was evacuated to 127 mm Hg and steam was then entered. The required temperature was normally attained within 30 to 60 seconds. Steaming was then continued for the predetermined time after which the autoclave was evacuated, the door unclamped and the yarn removed.

The yarn was steamed for one cycle only in all the cases. After steaming, the yarn was removed from the cop and rinsed in cold water to remove the fugitive dye which was present on the wool and which interfered with the determination of the various dyes used in this study. Preliminary investigations showed that there was little difference between the dye affinities of the wool from the outside and the inside of the cops. In all cases, however, the yarn was homogenised before a sample was taken for dyeing studies. The yarn was then dyed at a wool to liquor ratio of 1:70 with one *per cent* dye (on mass of wool) in perforated baskets in an Ahiba laboratory dyeing apparatus. All the dyeings were carried out according to the methods recommended for pale shades by the various dye manufacturers. Some of the dyeing recipes which were used, are given in Appendix 1. Dyeing commenced at 40°C and the temperature of the bath was raised to the boil at a rate of 1°C/minute. The dyeing was continued at the boil for 120 minutes. The rate of exhaustion of the dye was determined spectrophotometrically.

For a visual evaluation of the effect of setting on the dye exhaustion, and therefore the depth of shade of certain dyes, yarns which had been set under different conditions were knitted into fabrics on a Lawson Fibre Analysis Knitter. The various fabrics produced were then sewn together and dyed on a winch. After dyeing the different fabrics were ranked according to their depth of shade by 10 observers.

For the determination of the effect of steaming on snarling twist, yarns produced at SAWTRI were taken directly from the spinning machine, conditioned for 24 hours at 65 *per cent* RH and 20°C, followed by setting. The characteristics of these yarns are also given in Table I. Snarling twist before and after setting was

**TABLE I**  
**CHARACTERISTICS OF THE YARNS USED IN THIS INVESTIGATION**

YARN	TEX	SNARLING TWIST (t.p.m)
Commercial yarn	37,1	90
SAWTRI spun yarn	15,5	63
"     "     "	18,0	91
"     "     "	21,0	76

TABLE II

THE DIFFERENCE BETWEEN THE RATES OF EXHAUSTION OF VARIOUS DYES ONTO UNSET WOOL AND WOOL SET AT 120°C FOR 40 MINUTES

TYPE	DYE	DIFFERENCE BETWEEN PERCENTAGE EXHAUSTION ONTO UNSET AND SET WOOL	AVERAGE (%)
1:1 Metal Complex	Acid Red 195	3	16
	Acid Violet 56	11	
	Acid Yellow 99	13	
	Acid Blue 158A	15	
	Acid Orange 74	23	
	Acid Green 12	24	
	Palatine Fast Blue RRN (B.A.S.F.)	26	
1:2 Metal Complex	Cibalan Blue FBL (Ciba-Geigy)	2	7
	Acid Orange 87	3	
	Acid Brown 225	3	
	Acid Brown 369	3	
	Acid Green 43	4	
	Acid Red 217	7	
	Acid Red 209	12	
	Acid Violet 66	20	
Acid Leveling	Acid Yellow 15	1	8
	Acid Blue 72	2	
	Acid Red 43	3	
	Acid Violet 1	8	
	Acid Orange 10	12	
	Acid Green 5	13	
Acid Milling	Acid Green 9	14	13
	Acid Violet 51	1	
	Acid Blue 127 (similar)	7	
	Acid Red 138	8	
	Acid Green 27	9	
	Acid Yellow 72	10	
	Acid Blue 120	10	
	Acid Orange 33	11	
	Acid Yellow 44	12	
	Acid Red 134	12	
	Acid Violet 34	17	
	Acid Red 85	18	
	Acid Brown 49	18	
	Acid Blue 92	24	
Acid Green 25	25		
Reactive	Reactive Red 100	2	16
	Reactive Red 22:1	6	
	Reactive Red 51	8	
	Reactive Blue 94	10	
	Reactive Orange 16:1	10	
	Reactive Red 38	14	
	Reactive Yellow 39	14	
	Acid Yellow 127*	15	
	Hostalan Brilliant Yellow C (Hoechst)	21	
	Acid Red 253*	21	
	Reactive Orange 29	22	
	Reactive Blue 69	22	
	Reactive Red 40	25	
	Reactive Yellow 69	28	

\*These dyes are classified as being fibre-reactive dyes.

determined according to the method suggested by WIRA by counting the number of turns in a loop of yarn of one metre girth<sup>(1)</sup>. The snarling twist was expressed as the number of turns per metre in the resultant half-metre loop of yarn (i.e. twice the number of turns counted in the loop itself).

The urea-bisulphite solubility (U.B.S.) of the samples was determined according to the method suggested by the IWTO<sup>(10)</sup>. The breaking strength of the yarns was determined in the usual manner. The degree of whiteness of the undyed knitted fabrics was determined on a Zeiss Elrepho apparatus, using the formula suggested by Berger<sup>(11)</sup>.

## RESULTS AND DISCUSSION

Approximately 50 different commercially available dyes were used in this investigation. The dyes were selected from the 1:1 metal complex, 1:2 metal complex, acid milling, acid levelling and reactive types. Initially, the rates of exhaustion of all the dyes were determined using wool which had not been set, as well as wool set under very drastic conditions, namely for 40 minutes at 120°C. Such severe conditions are not usual for the setting of wool, but were selected to accentuate the effect of setting on the rates of exhaustion of the dyes.

There are several possible ways in which the effect of setting on the affinity of wool for a dye can be demonstrated. In this investigation the difference between the percentage of dye exhausted onto the set wool, and the percentage of dye exhausted onto the unset wool was determined for each dye, and the results are given in Table II. This absolute difference, for convenience sake, will be defined as the "setting sensitivity index" (SSI). It gives an indication of the effect of setting on the rate of exhaustion of a specific dye onto wool. The greater the effect of the setting treatment, the higher the setting sensitivity index. The values given in Table II are the largest differences observed at any stage of the dyeing and are not necessarily those obtained at the completion of dyeing.

In most cases the dye exhaustion curves followed a similar pattern, with the rate of exhaustion higher in the case of the set wool than in the case of the unset wool. Typical examples of the dye exhaustion curves obtained are shown in Figures 1 and 2. The effect of setting on the rate of exhaustion of the dye became noticeable at an early stage of dyeing, normally within 15 minutes after dyeing had commenced. The difference between the rates of exhaustion increased up to a certain stage and then remained fairly constant until dyeing was concluded (Figure 1). In a few cases, however, the difference seemed to increase at the initial stages of dyeing, reaching a maximum value, after which it decreased again (Figure 2). The largest difference observed at any stage of the dyeing is given in Table II.

The results in Table II show several interesting points. In practically all cases setting *increased* the rate of exhaustion of the dye. This was so for all the different types of dyes studied. In many cases the setting sensitivity index was more than

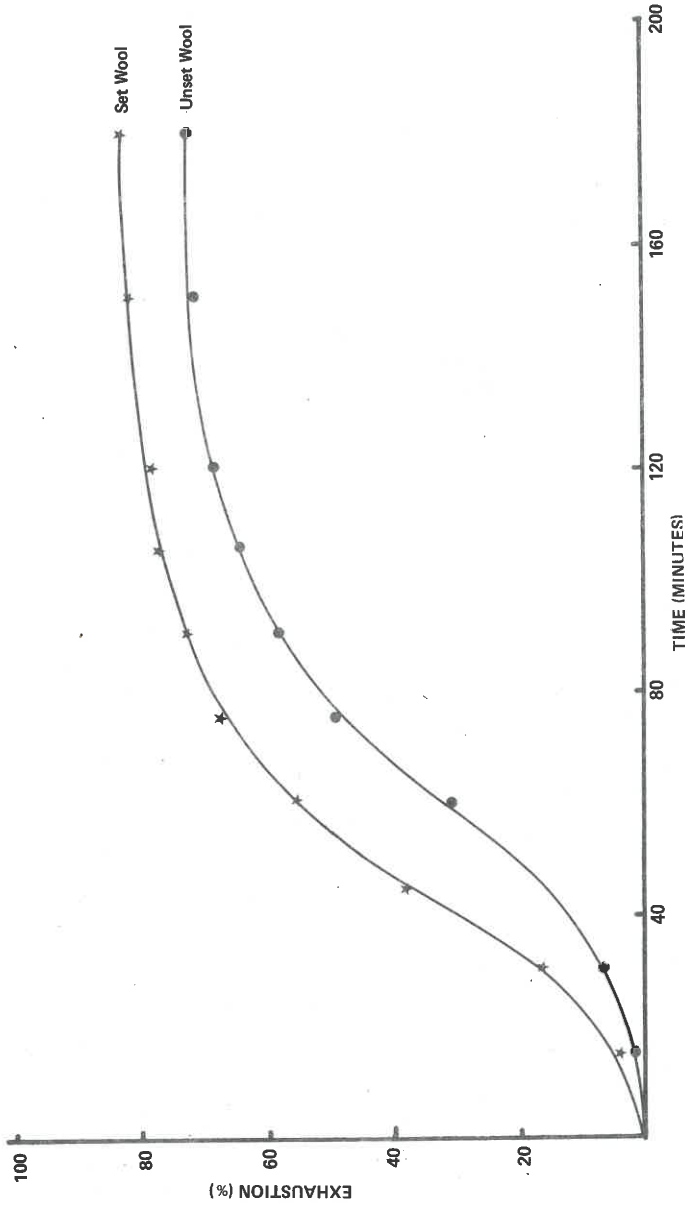


FIGURE 1

The rates of exhaustion of Reactive Blue 94 onto unset wool and wool which had been set at 120°C for 40 minutes



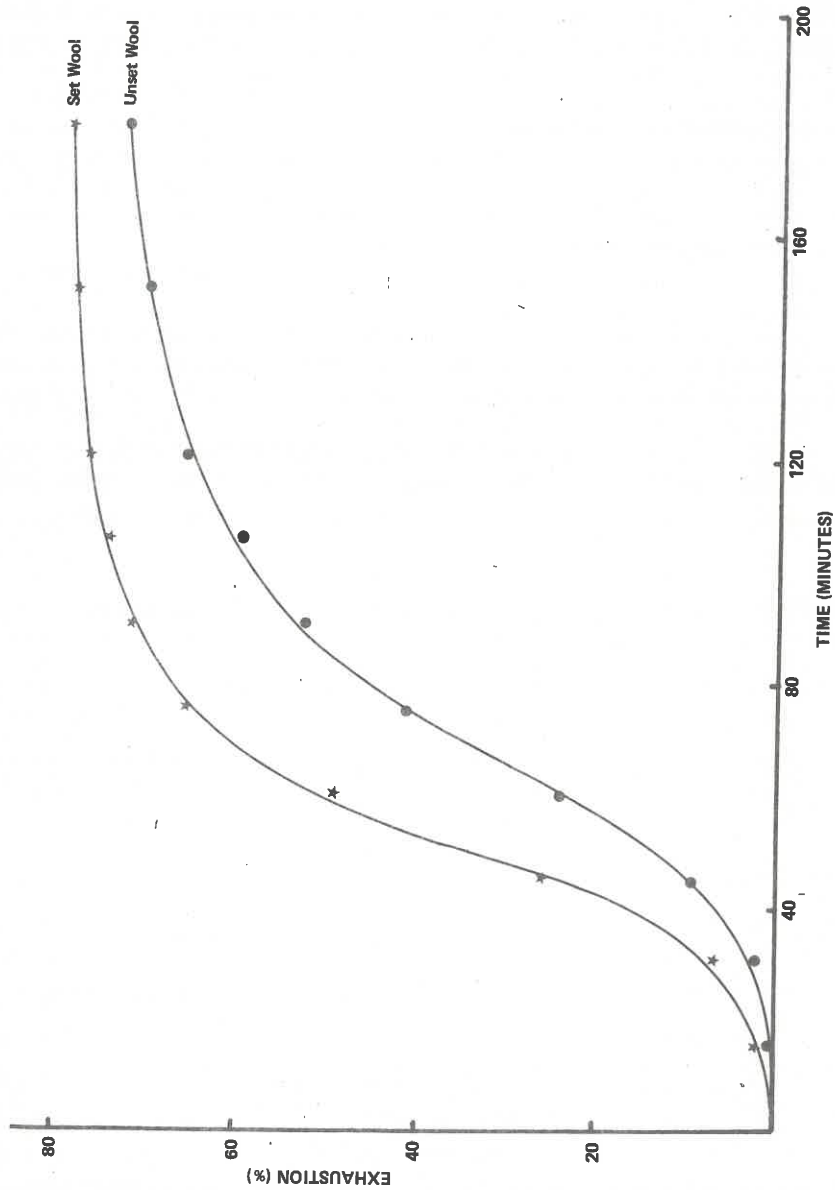


FIGURE 2

The rates of exhaustion of Hostalan Brilliant Yellow C onto unset wool and wool which had been set at 120°C for 40 minutes

20 per cent, which was to be expected, because of the very severe setting conditions, which probably caused some damage to the wool. Surprisingly, however, it was found that the rate of exhaustion of *certain* dyes was *not* affected significantly by the setting treatment. For each of the different types of dyes studied, certain dyes were found with a setting sensitivity index of less than 5 per cent. Such dyes could be usefully employed in cases where differences in the degree of setting of the wool were anticipated.

Approximately 24 per cent of all the dyes studied, had setting sensitivity indices of less than 5 per cent, and 23 per cent greater than 20 per cent. The 1:2 metal complex and acid levelling dyes seemed the least sensitive to setting, while the 1:1 metal complex and reactive dyes seemed most sensitive.

Some further studies were then carried out by setting wool yarn for various periods at 120°C. The set wool as well as the unset wool were then dyed with some of the dyes and the setting sensitivity indices were subsequently calculated. These are shown in Figure 3 as a function of the time of setting. It can be seen that the setting sensitivity index increased rapidly with an increase in the time of setting. Some of the dyes were found to be extremely sensitive to setting at 120°C. The Reactive Yellow 69 and Reactive Red 40 dyes, for example, had setting sensitivity indices greater than 10 per cent after a setting time of only 2 minutes. In most cases

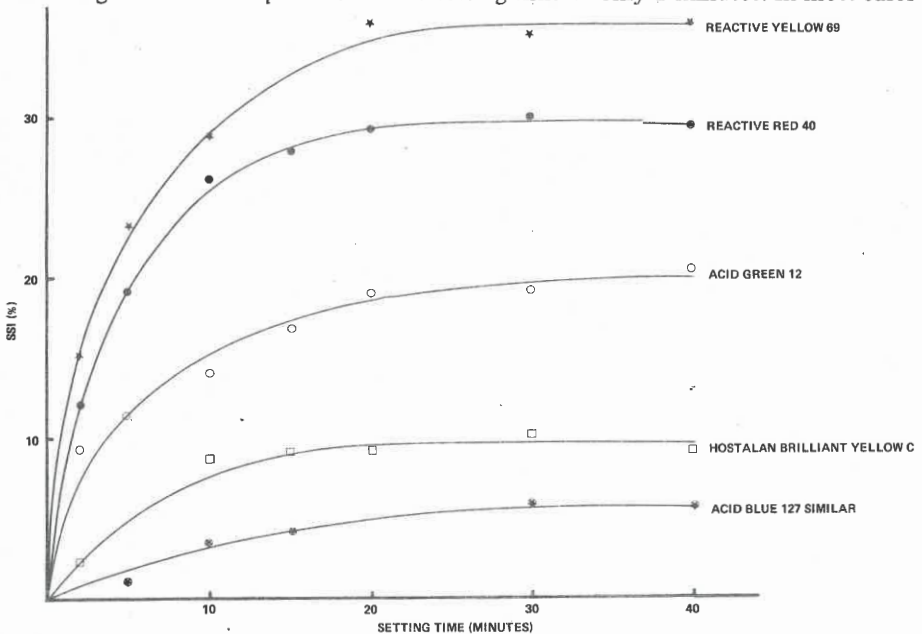
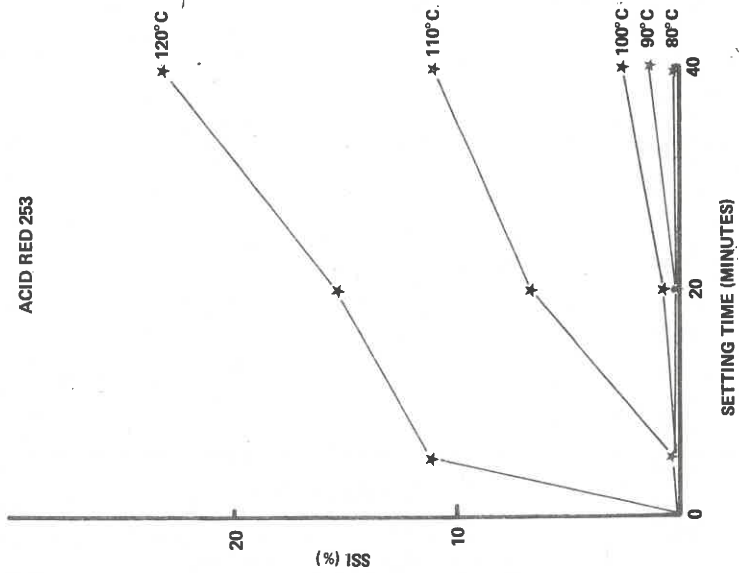
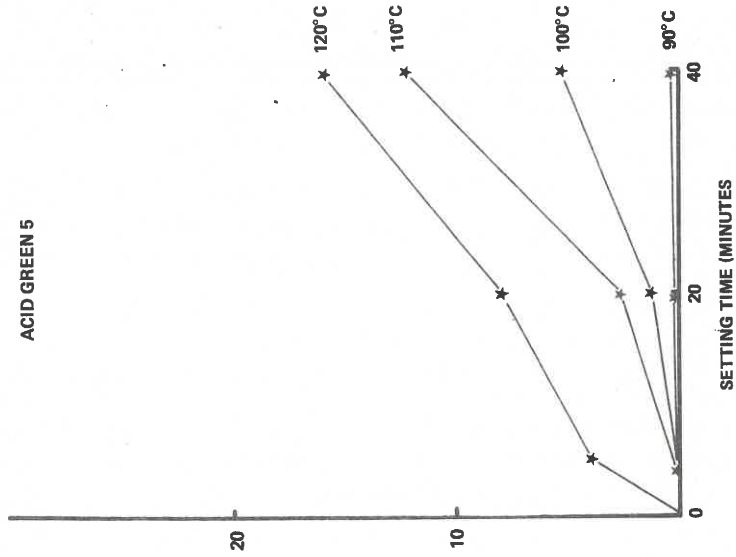


FIGURE 3

The effect of time of setting (at 120°C) on the setting sensitivity indices of certain dyes



**FIGURE 4**  
The effect of temperature and time of setting on the setting sensitivity indices of certain dyes

the setting sensitivity index increased up to a setting time of about 20 minutes, after which it seemed to level off. Figure 3 also clearly illustrates that the rates of exhaustion of the dyes were affected to different degrees by the setting treatment.

Figure 4 shows the effect of setting at various temperatures on the setting sensitivity index of certain dyes (Acid Green 5 and Acid Red 253). Setting at 80°C had no effect on the setting sensitivity index of the dyes, up to a setting time of 40 minutes. Setting at 90°C had practically no effect on the setting sensitivity index, as did setting at 100°C for 20 minutes or less. When the time of setting was 40 minutes (100°C), the setting sensitivity index increased slightly but it is not clear, however, whether these differences are of any practical importance. Setting at 110°C, for 20 minutes or more, generally increased the setting sensitivity index significantly, while setting for 5 minutes or less did not have any effect. Setting at 120°C for 5 minutes, however, resulted in high setting sensitivity indices.

The effect of setting on the degree of whiteness of wool is shown in Fig. 5. The results agree with those of Barker<sup>(12)</sup>. Setting at 100°C only slightly affected the degree of whiteness of the wool. When the temperature of setting was increased to 110°C, however, the degree of whiteness of the wool decreased significantly. When the temperature was further increased to 120°C, the wool yellowed to an

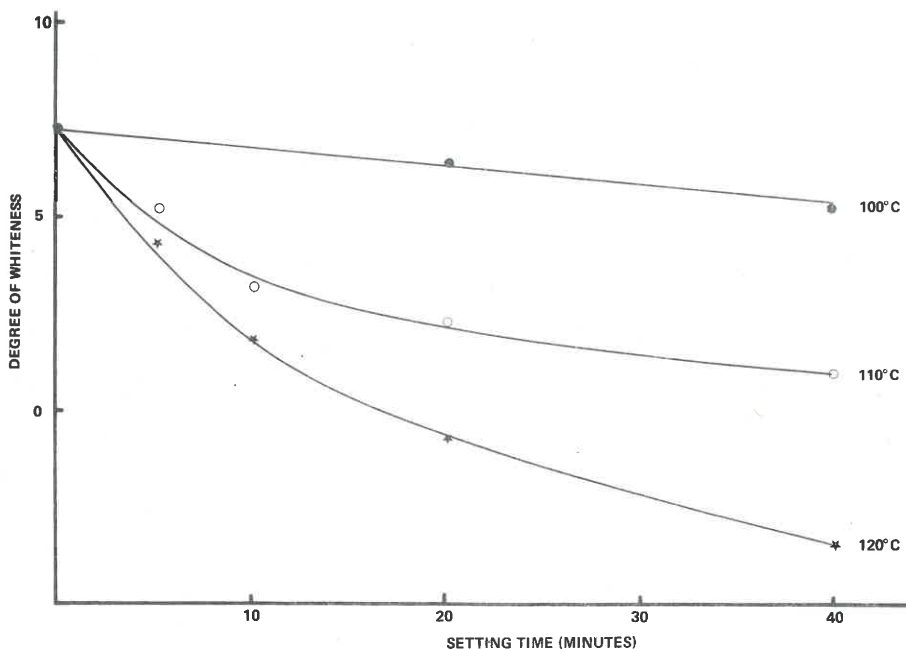


FIGURE 5

The effect of setting on the degree of whiteness of wool

even greater extent. The degree of whiteness of the wool generally decreased when the time of setting was increased. Figure 5 shows that relatively mild setting conditions, such as a treatment for 10 minutes at 110°C, resulted in a significant decrease in the degree of whiteness of the wool.

Five of the most sensitive dyes (as far as the rate of exhaustion onto set wool is concerned) were then selected and used to dye wool fabrics knitted from yarns which had been set under various conditions. The various fabrics were then ranked according to the depth of shade by 10 observers and the results obtained were subjected to statistical analysis. These are given in Tables III and IV. Table III gives the coefficient of concordance of the ranking of the various fabrics by the different observers, and shows that they were consistent in their ranking of the fabrics. The coefficient of concordance (W) is highly significant (better than 99,5%) in all cases. The accuracy of assessing the different shades was examined by use of factorial analysis of variance techniques. The results obtained are given in Table IV. The following conclusions can be drawn from this Table:

(i) The fabrics dyed with Reactive Red 40, Reactive Yellow 69 and Hostalan Brilliant Yellow C were ranked in the expected order, that is, becoming darker with an increase in the time or temperature of setting. In the case of Hostalan Brilliant Yellow C, however, the observers could not distinguish between the unset fabric and the fabric set at 110°C for 10 minutes.

**TABLE III**  
**THE COEFFICIENT OF CONCORDANCE OF THE RANKING OF THE VARIOUS FABRICS**

DYE	COEFFICIENT OF CONCORDANCE*
Acid Violet 66	0,337
Hostalan Brilliant Yellow C	0,397
Reactive Blue 94	0,485
Reactive Yellow 69	0,827
Reactive Red 40	0,890

\*A value of 0,229 denotes a statistical significance of 99,5%

TABLE IV

## MEAN VALUE TABLES FOR THE RANKING OF THE VARIOUS FABRICS

DYE	MEAN VALUES*					6 <sup>v</sup>	( $\bar{x}$ ) MINIMUM DIFFERENCE BETWEEN ANY TWO MEAN VALUES FOR THOSE VALUES TO BE JUDGED SIGNIFICANTLY DIFFERENT
	Control	Setting time (mins)			Setting temperature (°C)		
		10	20	40			
Acid Violet 66	2,8	3,6	6,0	5,0	110	1,32	1,25
		5,4	4,8	1,8	120		
Hostalan Brilliant Yellow C	1,4	2,1	3,9	5,3	110	1,27	1,19
		3,6	5,1	6,0	120		
Reactive Blue 94	1,7	3,3	3,9	3,0	110	1,36	1,27
		5,9	5,0	6,1	120		
Reactive Yellow 69	1,1	2,3	2,6	5,0	110	0,71	0,67
		4,4	5,2	7,0	120		
Reactive Red 40	1,0	2,0	2,7	4,6	110	0,48	0,45
		4,7	6,1	6,8	120		

\*Higher values mean darker shades

(ii) For Reactive Blue 94 the time of setting appeared to have no effect on the depth of shade, but the fabrics set at the higher temperatures were judged to be darker.

(iii) The differences shown with Acid Violet 66 did not follow any expected trend. The observers could, in other words, not rank the fabrics correctly in any order of increasing time or temperature of setting. This is rather surprising, since the dye exhaustion studies showed this dye to be fairly sensitive to setting conditions. Several of the observers did mention, however, that the fabrics dyed with this dye were difficult to rank or evaluate.

The results show that different setting conditions lead to large differences in the shades of the dyed wool fabrics and confirm the results obtained in the dye exhaustion studies. It must be pointed out, however, that setting also decreased the degree of whiteness of the wool. Probably the colour of the wool also played a rôle in the ranking of the fabrics. It is difficult, however, to establish the relative contribution of the colour of the wool itself compared with that of the dye, when the fabrics were ranked.

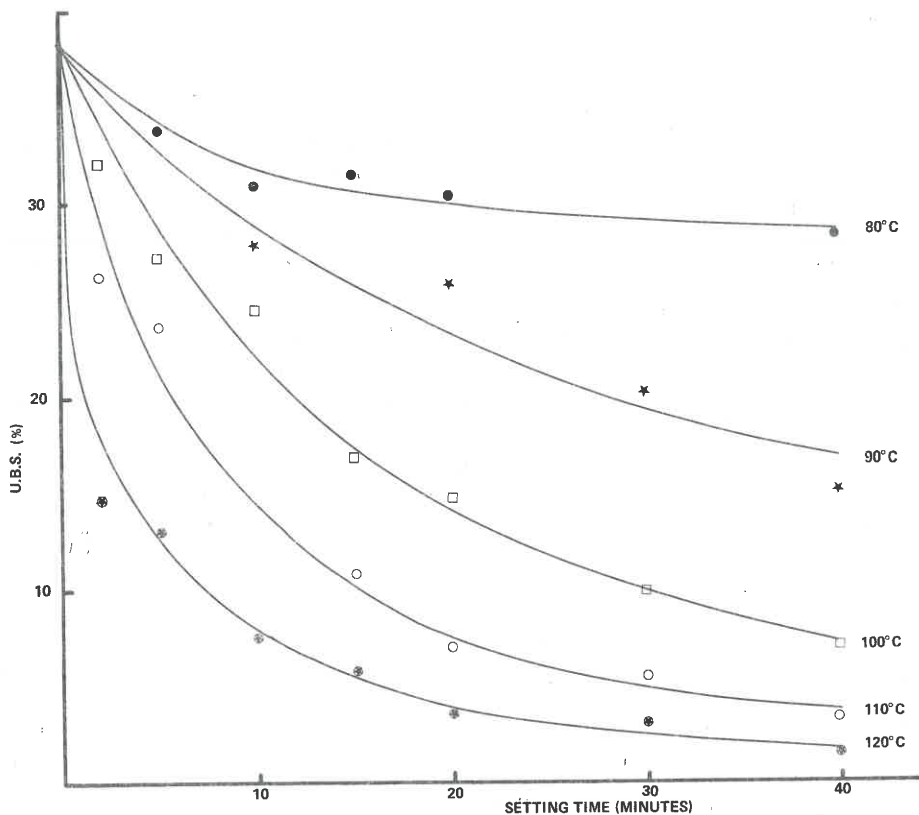
TABLE V

## THE EFFECT OF SETTING ON THE SNARLING TWIST OF YARN

SETTING CONDITIONS		SNARLING TWIST (t.p.m)		
Temperature (°C)	Time (mins)	Outside	Middle	Inside
90	5	8	14	18
90	10	6	12	10
100	5	6	14	18
100	10	6	6	6

The effect of setting on the urea-bisulphite solubility (U.B.S.) of wool is shown in Figure 6. The results are in agreement with the work of other research workers<sup>(2, 9)</sup> and show that the U.B.S. of the wool is extremely sensitive to setting treatments. The U.B.S. decreased when the temperature or time of setting was increased. The U.B.S. of the wool seems to be more sensitive to setting than any other parameter investigated and very mild setting conditions, such as treatment at 80°C or 90°C, resulted in a decrease in the U.B.S. of the wool. Setting at 110°C for 5 to 10 minutes, which are the conditions sometimes used in practice for the setting of wool yarn, reduced the U.B.S. of the wool by almost 50 per cent.

The effect of different setting conditions on the snarling twist of certain yarns was investigated subsequently. The three yarns spun at SAWTRI were set on the spinning cops 24 hours after they had been taken from the spinning machine. The results for the 18 tex yarns are given in Table V. Little difference was observed between these results and those pertaining to the other two yarns. The layer of yarn on the spinning cop had a thickness of about 10 mm and samples from the outside, middle and inside were taken for the determination of snarling twist.



**FIGURE 6**  
The effect of setting on the urea-bisulphite solubility of wool

There are no standards available for yarn liveliness but WIRA suggested that yarns with a snarling twist of less than 50 t.p.m would perform satisfactorily during processing<sup>(1)</sup>. Yarns with a snarling twist of less than 10 t.p.m are normally classified as "dead". The results (Table V) clearly show that very mild setting conditions reduced the snarling twist of the yarn to acceptable levels. In all the cases the snarling twist of the yarns was less than 20 t.p.m and in some cases (10 minutes at 100°C) "dead" yarns with a snarling twist of less than 10 t.p.m were obtained. Setting of yarn at a temperature of 100°C, or lower, has been recommended by several other research workers<sup>(2, 5, 6)</sup>. Some further studies on the effect of different setting conditions on the snarling twist of yarn on large cones will be carried out and will be reported on at a later stage.



**TABLE VI**  
**THE EFFECT OF SETTING ON THE BREAKING STRENGTH AND**  
**EXTENSION OF THE YARNS**

	SETTING TEMPERATURE (°C)	BREAKING STRENGTH (gf) AND EXTENSION (%) OF YARN*			
		Setting Time (Mins)			
		5	10	20	40
18 Tex Yarn	90	116 (12,8)	113 (11,4)	117 (12,0)	122 (14,2)
	100	114 (12,4)	116 (12,4)	112 (10,6)	113 (11,8)
	110	116 (11,8)	102 (10,2)	101 ( 9,6)	105 ( 9,6)
	120	114 (11,2)	108 ( 9,8)	108 ( 9,0)	95,2 (7,6)
	Untreated	110 (13,1)			
39 Tex Yarn	90	306 (23,8)	277 (18,4)	317 (20,4)	281 (19,9)
	100	281 (17,0)	306 (25,4)	286 (21,0)	289 (21,4)
	110	283 (21,2)	280 (20,0)	269 (18,6)	244 (16,6)
	120	276 (19,4)	275 (17,2)	269 (18,6)	259 (16,4)
	Untreated	294 (22,4)			

\* Breaking extension is given in parenthesis.

Finally the effect of setting on the breaking strength and extension of the yarns is given in Table VI. There appeared to be a tendency for the breaking strength and extension to decrease as the setting conditions became more severe. This indicates, once again, that as low a temperature and as short a time as possible should be employed for the setting of yarns.

### SUMMARY

The rates of exhaustion of approximately 50 different commercial dyes onto unset wool as well as autoclave set wool were determined. In practically all cases setting increased the rates of exhaustion of the dyes. Some dyes were affected to a much greater extent than other dyes. The affinity of wool for certain dyes was, however, not significantly affected by setting. The 1:2 metal complex and acid levelling dyes seemed to be the least sensitive to setting, while the 1:1 metal complex and reactive dyes seemed most sensitive.

The effect of different setting conditions on the rates of exhaustion of certain dyes (Acid Green 5 and Acid Red 253) was also investigated. Setting at 80°C and 90°C for various periods up to 40 minutes, did not affect the rates of exhaustion. The same applied to setting at 100°C, except that a setting time of 40 minutes seemed to increase the rates of exhaustion slightly. Setting at 110°C for 5 minutes, or less, had no effect, while a setting time of 20 minutes or more increased the rates of exhaustion significantly. Setting at 120°C increased the rates of exhaustion of the dyes significantly. The rates of exhaustion of the dyes generally increased when the time of setting was increased.

The urea-bisulphite solubility (U.B.S.) of the wool seemed to be more sensitive to setting than was the dye affinity. The U.B.S. of the wool decreased when the temperature or time of setting was increased. Setting under very mild conditions (80°C or 90°C) resulted in a decrease in U.B.S. of the wool. When the wool was set at 110°C for 5 to 10 minutes, the U.B.S. was reduced by almost 50 per cent.

Furthermore the results show that the breaking strength and extension of the yarn decreased as the setting conditions became more severe. The degree of whiteness of the wool also decreased when the temperature or time of setting was increased.

The effect of different setting conditions on the snarling twist of the yarn on cops was also studied. It was found that very mild setting conditions were sufficient to reduce the snarling twist of the yarn to acceptable levels.

### ACKNOWLEDGEMENTS

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### THE USE OF PROPRIETARY NAMES

The fact that products with proprietary names have been mentioned in this report in no way implies that there are not others which are as good or better.

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## APPENDIX

### FIGURE 1:

1% Reactive Blue 94  
1% Acetic Acid (60%)  
10% Sodium Sulphate  
1% Avolan RE (Bayer)

### FIGURE 2:

1% Hostalan Brilliant Yellow C  
2% Ammonium Acetate  
pH adjusted to 5,5 with Acetic Acid (60%)  
1,5% Albegal B (Ciba-Geigy)

### FIGURE 3

1% Reactive Yellow 69  
1% Acetic Acid (60%)  
10% Sodium Sulphate  
1% Avolan RE (Bayer)  
  
1% Reactive Red 40  
1% Acetic Acid (60%)  
10% Sodium Sulphate  
  
1% Acid Green 12  
1% Sulphuric Acid  
  
1% Acid Blue 127 similar  
2% Ammonium Sulphate  
1% Albegal A (Ciba-Geigy)

### FIGURE 4

1% Acid Red 253  
10% Sodium Sulphate  
4% Ammonium Sulphate  
1% Albegal B (Ciba-Geigy)  
  
1% Acid Green 5  
10% Sodium Sulphate  
pH adjusted to 2 with formic acid

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