

**Sequential Oxidative/Reductive Bleaching of Wool in a Single Bath:**

**Determination of Optimum Conditions and Study of Fabric Handle for Worsted**

**Challis<sup>1</sup>**

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## **ABSTRACT**

**The properties of worsted challis fabric were examined after bleaching by conventional alkaline peroxide bleach and by variations of the Agricultural Research Service (ARS) two-step, single-bath sequential oxidative/reductive process. Whiteness and Yellowness Indices and mechanical properties of fabrics bleached by the conventional process with 22g/L (20mL/L) 30% hydrogen peroxide at 50C and 60C were compared with those of fabrics bleached by the ARS process with (a) 16g/L; or (b) 8g/L in Step 1 followed by the addition of thiourea (70% of the weight of peroxide) in Step 2; or (c) 22g/L peroxide in Step 1 followed by modifying the baths to reach an effective peroxide level of either 16g/L or 8g/L before the addition of thiourea. ARS-bleached fabrics were whiter or equal to the peroxide-bleached fabrics bleached within the same time and temperature limits. ARS-bleached challis had retained original strength but when compared to the peroxide-bleached challis, lost 11% specific stress, with only slight changes in elastic moduli and % strain at peak stress. Evaluation of fabric hand by the Kawabata Evaluation System (KES-F) showed an increase in Shinayakasa, indicating greater softness, flexibility, and smooth feeling for the ARS-bleached fabrics.**

## **Introduction**

In the United States, domestic wool provides about a third of the needs of the American textile industry. American wool is mainly a by-product of the meat industry, and as such it often contains an excessive amount of stained and pigmented fibers. As a result, its perceived quality and its price are significantly lower than its imported (mainly Australian) counterpart. The objective of this research is to improve the value of domestic wool, and in particular to develop new technology to bleach stained and pigmented fibers.

The most effective bleaching regimens for stain incorporate both an oxidative and a reductive bleaching step and are referred to as "full" bleaching. Such full bleaching is normally a two- or three- step process carried out in separate baths. Initial oxidative bleaching normally makes use of hydrogen peroxide. Subsequent reductive bleaching uses such agents as dithionite, sodium formaldehyde sulfoxylate, or thiourea dioxide (1).

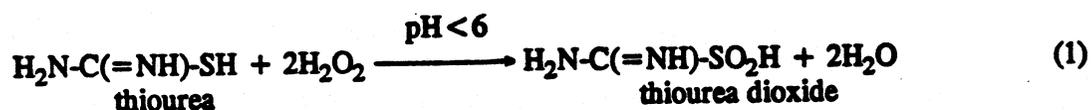
In a series of recent reports (2-6), patents and patent applications (7-13), we have described the development of new approaches to full bleaching that integrate the processes into single bath procedures (Fig. 1).

By conventional processes, initial bleaching with hydrogen peroxide is interrupted by quenching. Residual peroxide can simply be destroyed catalytically and a subsequent reductive bleaching carried out by addition of reducing bleach. In the ARS process, residual peroxide is best utilized in a quick chemical reaction that converts the bath into a reductive medium. When thiourea is added under controlled conditions to the peroxide bath, thiourea dioxide is produced. It then hydrolyzes to sulfinate—the reductive bleaching species (2,4-8,11,13).

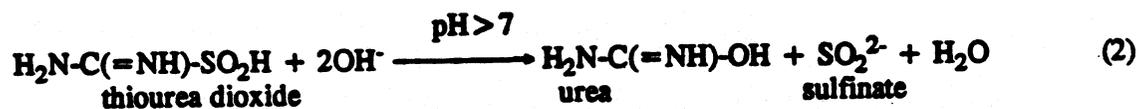
Bleaching for black-hair removal typically is done by initial mordanting with ferrous sulfate, then controlled rinsing to eliminate all iron except that which is selectively adsorbed onto the black fibers. Then, hydrogen peroxide is added, black hairs are bleached by a free-radical mechanism induced by

adsorbed iron, and overall stain is bleached concomitantly (14). Here again, we have found that residual peroxide may be utilized by conversion of the bleach bath to a reducing medium by controlled addition of thiourea (Fig. 2). This process is more effective for bleaching stain that could have been obscured by the pigmentation. It also eliminates traces of any orange-colored ferric species (from the mordanting) by reduction to soluble ferrous ion (3,4,9-11,13).

A study (6) by  $^{13}\text{C}$  NMR of the chemistry of the reductive bleaching process shows that thiourea reacts initially with hydrogen peroxide to form thiourea dioxide (Equation 1):



Thiourea dioxide in turn undergoes hydrolysis when the pH of the bath is adjusted to 7-8. It is at this step that the redox potential becomes negative, indicating formation of a reductive medium. NMR shows that thiourea dioxide is hydrolyzed to urea and presumably sulfinate ion, the true reductive species (Equation 2):



Sulfinate in turn acts on the wool and is oxidized to sulfate during the bleaching period (Equation 3):



(17) Peters, R.H., *Textile Chemistry, Impurities in Fibres; Purification of Fibres*, Elsevier Publishing Company, Amsterdam, Vol. II, 1967, p. 279.

(18) Zhan, H., Chemical Test Methods in Wool Processing, *Wool Science Review* Vol. 32, 1967, p. 1-15.

(19) Cardamone, J.M., and Marmer, W.N., *Textile Chemist and Colorist*, in press.

- (a) By varying the thiourea concentration and bleaching times, maximum reduction potential and whiteness could be achieved—after one hour at 60C in Step 1—by the addition of thiourea in Step 2 in amounts at least 70% o.w. neat peroxide with a pH adjustment to 7.4 and 25 minutes of subsequent reductive bleaching.
- (b) By varying the time of oxidative bleaching in Step 1 from 40 to 60 min, followed by 25 minutes of reductive bleaching in Step 2 with 6.15g/L thiourea, (all at 60C) an improved whiteness of 8 WI units could be achieved over the results from 65 to 85 minutes of oxidative bleaching (Step 1) alone.
- (c) Using the same conditions as in (b), increasing the bath temperature from 60 to 70C increased whiteness by 3 WI; decreasing the bath temperature from 60 to 55C decreased whiteness by 2 WI.

For optimum bleaching and minimal fiber damage we investigated the use of reduced amounts of peroxide and concomitantly reduced amounts of thiourea. Thus, we extended the earlier studies to investigate the efficiency of bleaching when the initial peroxide concentration in Step 1 is reduced from 22g/L to 8g/L, 16g/L, or 12g/L. We also investigated corresponding "modified" bleach baths where the original 22g/L peroxide was decreased to 8g/L or 16g/L at the end of Step 1 by discarding a portion of the bleach bath. This allowed a lesser amount of thiourea to be needed for running Step 2. We carried out these reactions at 50C and 60C (and 55C in the case of 12g/L).

Further mechanistic studies also were carried out using  $^{13}\text{C}$  NMR solution spectroscopy and  $^{13}\text{C}$ -labelled thiourea to follow the conversion of the oxidative bath to the reductive bath.

## Experimental

### Materials

Thiourea<sup>2</sup>, thiourea dioxide (formamidinesulfonic acid), and sodium pyrophosphate (TSPP) decahydrate were obtained from Aldrich Chemical Co.<sup>3</sup>, Milwaukee, WI. Hydrogen peroxide was a 30% (w/w) aqueous solution obtained from Mallinckrodt, Inc., Paris, KY. Avolan UL-75 amphoteric wetting agent was provided by Mobay Corp., Pittsburgh, PA. Worsted wool challis, fabric count  $48 \times 44$ , fabric weight 3.7oz/yd<sup>2</sup> (warp, 2/50 and weft, 50s), fabric thickness 0.78 mm (at 0.5gf/cm<sup>2</sup>) was purchased as 530NC from Testfabrics (TF), Inc., Middlesex, NJ. It was received already mildly scoured, rinsed, decatized, but not carbonized.

### ARS Bleaching Technique

*Step 1, Oxidative Bleaching:* Bleaching was carried out at a liquor ratio of 30:1, typically using 10g of woolen fabric and 300mL of bleaching medium for each of six beakers in an Ahiba Texomat apparatus (Ahiba A.G., Dietlikon, Zurich, Switzerland). Wool was added at ambient temperature to an alkaline bleach bath of the following composition: 30% aqueous hydrogen peroxide (8.0g/L, equivalent to 2.4g/L neat hydrogen peroxide), tetrasodium pyrophosphate decahydrate (TSPP; 2.0g/L), Avolan UL-75 (0.167g/L). The pH of the bleach bath initially was 9.1. The bath temperature was increased at a rate of 1C/min to 60C (140F), and oxidative bleaching was allowed to

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<sup>2</sup>Although thiourea is a cancer-suspect agent, it is easily handled with care and is completely consumed upon reaction with hydrogen peroxide.

<sup>3</sup>Reference to a particular brand or firm name does not constitute endorsement by the U.S. Department of Agriculture over others of a similar nature not mentioned.

proceed for 1 hr from the addition of wool to the bath. At the end of this stage, the pH of the bleach bath had decreased somewhat (to 8.2-8.7).

*Step 2, Reductive Bleaching:* The bath, while still 60C, was acidified to a pH of 5.0-5.5 with acetic acid (56%; approximately 3mL/L). Then thiourea was added; 1.68g/L would be equivalent to 70% of the *initial* hydrogen peroxide, but titration of *residual* peroxide would probably show about a 10% loss by this time, and a concomitant adjustment in the amount of thiourea would be appropriate (though not necessary). Ten minutes was allowed for reaction with residual hydrogen peroxide in the bath (to form thiourea dioxide). Then sufficient aqueous ammonia was added to adjust the pH to 6.8-7.2 to obtain the plummet in potential to -640mV to carry out reductive bleaching. Reductive bleaching was allowed to proceed for an additional 25 minutes at 60C.

For bleaching with 12g/L and 16g/L peroxide levels, the bath amounts were proportional quantities. For modified ARS bleach baths, Step 1 with 22g/L peroxide was completed and a proportional volume of the bath was discarded and replaced with water to lower the concentration to 8, 12, or 16g/L before Step 2.

### **Variations in Bleaching for Optimum Conditions**

The various conditions for ARS bleaching are found in Table I. The system of nomenclature, such as "22/8T," indicates in abbreviated form the g/L of 30% aqueous peroxide in the initial step ("22"), followed by the (sometimes reduced) amount of peroxide in the second step ("8") that is converted to a reductive bleach by addition of thiourea ("T"). The control fabrics (0/0, 0/0 (50°) were carried through both steps, but without contact with any bleaching medium.

**Table I. Bleaching Conditions, Optimization Runs on Wool Challis**

Woolen Challis	Step 1: Oxidative		Step 2: Reductive		T (C)
	[H <sub>2</sub> O <sub>2</sub> ] (g/L)	t (min)	[H <sub>2</sub> O <sub>2</sub> ] (g/L)	t (min)	
0/0	0	85	0	25	60
0/0 (50C)	0	85	0	25	50
22/22	22	85	0	25	60
22/22 (50C)	22	85	0	25	50
16/16T	16	60	16	25	60
16/16T (50C)	16	60	16	25	50
8/8T	8	60	8	25	60
8/8T (50C)	8	60	8	25	50
12/12T (55C)	12	60	12	25	55
22/16T	22	60	16	25	60
22/16T (50C)	22	60	16	25	50
22/8T	22	60	8	25	60
22/8T (50C)	22	60	8	25	50

Replications: There are three replications. Each replication represents 6 fabrics (10 grams each) with each treated in its own individual bath, liquor to fabric ratio, 30:1. All results represent mean values of 3 experiments (18 fabrics).

## **Measurements of Fabric Properties**

Whiteness index (WI; ASTM E-313; 3.387Z - 3Y) and yellowness index (YI; ASTM D-1925; 100 [1.277X - 1.06Z)/Y]) were measured on The Color Machine spectrophotometer (BYK Gardner).

Measurements were made using 360° circumferential illumination by a quartz halogen lamp at a color temperature of 2854K (CIE Source C illuminant, CIE Standard 2° observer) at a 45° angle from the sample's normal direction, with sample viewing at 0°.

## **Fabric Handle**

The Kawabata Evaluation System (KES-F) was used to examine the fabric's physical and mechanical properties, using weight, thickness, and tensile, bending, shearing, and compression data. For each bleaching condition, the average of three readings was recorded. Instrument settings were as follows:

Compression: rate of compressing, 0.02mm/s; maximum force, 50.0gf/cm<sup>2</sup>; area compressed, 2.0cm<sup>2</sup>/circle. Bending: rate of bending, 0.5cm<sup>-1</sup>/s; maximum curvative, ± 2.5cm<sup>-1</sup>; sample size, 1 × w, 15-20cm × 1cm. Shear: rate of shearing, 0.417mm/s; maximum shear angle, ± 8°; tension on sample, 10gf/cm. Sample size, 1 × w, 15-20cm × 5cm. Tensile: rate of extension, 0.2mm/sec; maximum tensile force, 500gf/cm; sample size, 1 × w, 15-20cm × 5cm. Fabric Weight (15-20cm<sup>2</sup> area) reported in mg/cm<sup>2</sup>.

## **Alkali-Solubility**

Alkali solubility of wool after bleaching was determined according to test procedure ASTM D1283-84, "Method of Test for Solubility of Wool in Alkali." Fabrics (1.0 gram each) were brought to constant dry weight before and after exposure to 0.1N sodium hydroxide solution at 65C for one hour. The resulting solutions were then filtered of their fabric residues through sintered glass of medium porosity by vacuum aspiration. Fabric residues were washed, dried, and weighed. The

degree of damage to wool was then estimated in terms of the loss in fabric weight as a percentage of the original weight of the sample.

### **Instron Testing**

Fabrics were measured in the wet state for specific stress, elastic modulus, and percentage strain at peak stress on an Instron Model 1122 Analyzer. Analyses were carried out according to ASTM 1682-64, "Raveled Strip Method" for wet specimens (section 17.2). A load cell, Instron 2511-103, 50lb load capacity with crosshead speed 200mm/sec, was used for these fabrics. Force to break was normalized to the fabric linear density (g/cm) with specific stress reported as Newtons cm/gram.

## NMR Spectroscopy

$^{13}\text{C}$ -NMR spectra were obtained on a Bruker MSL-300 instrument operating at 75.5MHz. All spectra were obtained with a 9 microsec (80C) pulse with a recycle time of 10 sec. Each spectrum was obtained from 66 8k-data-point scans. Temperature was controlled to within  $\pm 1\text{C}$ . We examined the 8 and 16g/L (Step 2) reactions at 50C and 60C and at pH ranges of 7.1-7.8 and 8.4-8.9. The procedure involved preparing 15mL stock solutions of 8.0g/L ( $1.1 \times 10^{-3}\text{M}$ ) peroxide and 16g/L ( $2.1 \times 10^{-3}\text{M}$ ) peroxide. The corresponding amounts of thiourea required were  $3.3 \times 10^{-4}\text{mol}$  and  $6.6 \times 10^{-4}\text{mol}$ , respectively, wherein the peroxide to thiourea molar ratio was 3.2 in each case. Aliquots (3mL) were taken for each NMR analysis.

## Results and Discussion

### Reaction Mechanism

The NMR spectrum of  $^{13}\text{C}$ -thiourea (Fig. 3a) was not stable in the presence of hydrogen peroxide at pH 4.5-5.5, but changed instead to the signal for thiourea dioxide (179.8ppm). When the pH then was raised to  $>7$ , complete reaction of the thiourea dioxide was seen, leaving only a signal for the reaction product, urea (164.7ppm). Under the mildest bleaching conditions (8g/L, 50C, pH 7.8), however, the thiourea dioxide signal remained pronounced along with the new signal for urea (Fig. 3b). Complete conversion to urea in that case was observed (Fig. 3c) only after wool was added and stirred into the bleaching solution.

These findings support the work of others who found that the decomposition of thiourea dioxide is controlled by the effect of temperature and time, with higher temperature causing greater decrease in thiourea dioxide in the absence of wool. In the presence of wool there was an obvious acceleration of the rate of decomposition of thiourea dioxide (15).

## Whiteness and Yellowness Indices

The average values for whiteness index (WI) and yellowness index (YI) for 18 replications for each bleaching condition are reported as observed values and have been analyzed for statistical differences. The estimated values used to formulate the graphs in Figs. 4 and 5 were derived from data specific to 5 points (8g/L at 50C and 60C, 16g/L at 50C and 60C, and 12g/L at 55C) using the method of least squares from the multiple regression equations 4 and 5 to describe the influence of temperature (T), peroxide concentration [H<sub>2</sub>O<sub>2</sub>], and the combined effects:

$$WI = 43.745 - 0.42T - 2.89 [H_2O_2] + 0.066T [H_2O_2] \quad (R^2 = 0.918) \quad (4)$$

$$YI = 13.04 + 0.123T + 0.973 [H_2O_2] - 0.022T [H_2O_2] \quad (R^2 = 0.900) \quad (5)$$

Those equations are predictive to estimate the effectiveness of bleaching at specific temperature and peroxide concentration. Overall the process is much more sensitive to temperature at higher peroxide concentrations.

## Fabric Handle

Evaluation of fabric handle by the Kawabata Evaluation System (KES-F) was made on three fabrics selected randomly from the 18 replications per bleaching condition. Each fabric was tested in the warp and weft directions. The most pronounced differences (Fig. 6, a-d) when conventionally-bleached and ARS-bleached fabrics are compared are in the parameters EM% (extension maximum at 500g/cm) in (a), 2HB (Bending Hysteresis) in (b), 2HG (Shearing Hysteresis at 0.5° shear angle) (c), and in 2HG5 (Shearing hysteresis at 5° shear angle) in (d). These parameters, KES characteristic values, are the main contributors to the KES hand expression, "Shinayakasa," which indicates softness, flexibility, and smooth feeling. Shinayakasa is a standard of hand evaluation for women's

thin dress fabric (16). Shinayakasa results are shown in Fig. 7: these objectively derived hand-ranking scores were compared to subjective panel scores for softness (Fig. 8) where control, 0/0 (50C), was assigned a score of 5 on a scale of 1-10 (10 being softest). Fig. 9 shows the correlations of bending hysteresis, 2 HB, with the panel scores.

Chemical damage from bleaching is reported (in terms of alkali solubility) in Table II below:

**Table II. Alkali Solubility of Bleached Worsted Challis**

Sample	Alkali Solubility (%)
0/0	10.3 ± 0.4
0/0 (50C)	9.7 ± 0.6
16/16T	19.7 ± 1.0
8/8T	13.3 ± 0.8
8/8T (50C)	17.0 ± 0.5
12/12T (55C)	14.6 ± 1.0
22/8T	19.9 ± 0.9
22/16T (50C)	14.6 ± 0.9
22/22	18.1 ± 1.0
22/22 (50C)	18.4 ± 0.5

The alkali solubility of undamaged wool has been reported as 12-13% and, for damaged wool, greater than 18% (17). By 30%, mechanical properties have deteriorated considerably (18). The values for alkali solubility of ARS- and conventionally bleached fabrics in Table II are within the range of minimal damage.

## **Mechanical Property Testing**

Results from Instron testing are given in Figs 10-12. Darker bars indicate whiteness (WI) and lighter bars indicate the mechanical property (specific stress, elastic modulus, and percent strain at peak stress, respectively). In each figure, the top set compares all bleachings (50 and 60C) shown to give equivalent or better whiteness than the conventionally bleached sample 22/22. The bottom set compares all 50C bleachings that gave equivalent or better whiteness than 22/22 (50C). All bars labelled with a common letter are not statistically different ( $p < .05$ ).

Mechanical properties of fabrics bleached by the various bleaching regimens are consolidated below. These results are grouped according to the degree of whiteness achieved. The specified mechanical properties may be loosely related to the following:

<b>Specific Stress:</b>	<b>Tensile strength</b>
<b>Elastic Modulus:</b>	<b>Resistance to extension</b>
<b>% Strain at Peak Stress:</b>	<b>Elongation at the breaking point</b>
<b>KES Shinayakasa:</b>	<b>Smooth feeling</b>
<b>KES Bending Hysteresis (2HB):</b>	<b>Resistance to return to position following bending</b>
<b>KES Shear Stiffness (G):</b>	<b>Stiffness to skewing within fabric plane</b>
<b>KES Shearing Hysteresis (2HG; 2HG5):</b>	<b>Resistance to return to position following skewing (at 0.5° or 5.0°)</b>
<b>KES Maximum Extension (EM%):</b>	<b>Extension under 500g load</b>

Note that the percentages given below are *relative to the values obtained from fabrics conventionally bleached* (85 minutes oxidative bleaching only; 60 or 50C; [30% aq H<sub>2</sub>O<sub>2</sub>] = 22g/L). From the loose descriptions above, the relative KES values *under* 100% for 2HB, G, 2HG, and 2HG5, and *over* 100% for EM% and Shinayakasa indicate improved softness characteristics.

Relative to conventional bleaching at 60° (22/22; WI = 33), the following observations were

made:

To achieve the *same degree of whiteness*, use the conditions of 12/12T (55C) (drop T to 55C; use only 12g/L H<sub>2</sub>O<sub>2</sub> (WI = 31)):

14% loss in specific stress\*.  
7% increase in elastic modulus.  
4% reduction in % strain at peak stress.  
KES Shinayakasa: 111%  
KES Bending Hysteresis 2HB: 75%  
KES Shear Stiffness G: 80%  
KES Shearing Hysteresis (0.5 deg) 2HG: 43%  
KES Shearing Hysteresis (5.0 deg) 2HG5: 48%  
KES Maximum Extension 500 g/cm, EM%: 128%

or use the conditions of 22/8T (50C) (drop T to 50C; start at 22g/L H<sub>2</sub>O<sub>2</sub>, but reduce to 8g/L (WI - 32)):

7% loss in specific stress\*.  
No change in elastic modulus.  
3% reduction in % strain at peak stress.  
KES Shinayakasa: 110%  
KES Bending Hysteresis 2HB: 78%  
KES Shear Stiffness G: 88%  
KES Shearing Hysteresis (0.5 deg) 2HG: 58%  
KES Shearing Hysteresis (5.0 deg) 2HG5: 58%  
KES Maximum Extension 500 g/cm, EM%: 112%

or use the conditions of 16/16T (keep T at 60C; use only 16g/L H<sub>2</sub>O<sub>2</sub> (WI = 36)):

11% loss in specific stress\*.  
5% increase in elastic modulus.  
No change in % strain at peak stress.  
KES Shinayakasa: 110%  
KES Bending Hysteresis 2HB: 73%  
KES Shear Stiffness G: 85%  
KES Shearing Hysteresis (0.5 deg) 2HG: 57%  
KES Shearing Hysteresis (5.0 deg) 2HG5: 57%  
KES Maximum Extension 500 g/cm, EM%: 126%

To achieve *better whiteness* using an ARS process, use the conditions of 22/16T (50C) (drop T to 50C; start at 22g/L H<sub>2</sub>O<sub>2</sub>, but reduce to 16g/L for Step 2 (WI = 36)):

6% loss in specific stress\*.  
No change in elastic modulus.  
4% reduction in % strain at peak stress.  
KES Shinayakasa: 106%  
KES Bending Hysteresis 2HB: 78%  
KES Shear Stiffness G: 86%  
KES Shearing Hysteresis (0.5 deg) 2HG: 53%  
KES Shearing Hysteresis (5.0 deg) 2HG5: 54%  
KES Maximum Extension 500 g/cm, EM%: 129%

or use the conditions of 22/8T (keep T at 60C; start at 22g/L H<sub>2</sub>O<sub>2</sub>, but reduce to 8g/L for Step 2 (WI = 37)):

9% loss in specific stress\*.  
No change in elastic modulus.  
3% reduction in % strain at peak stress.  
KES Shinayakasa: 106%  
KES Bending Hysteresis 2HB: 75%  
KES Shear Stiffness G: 87%  
KES Shearing hysteresis (0.5 deg) 2HG: 65%  
KES Shearing Hysteresis (5.0 deg) 2HG5: 63%  
KES Maximum Extension 500 g/cm, EM%: 130%

To achieve *the best whiteness* using an ARS process, use the conditions of 22/16T (keep T at 60C; start at 22g/L H<sub>2</sub>O<sub>2</sub>, but reduce to 16g/L for Step 2 (WI = 41)):

11% loss in specific stress\*.  
No change in elastic modulus.  
3% reduction in % strain at peak stress.  
KES Shinayakasa: 114%  
KES Bending Hysteresis 2HB: 77%  
KES Shear Stiffness G: 83%  
KES Shearing Hysteresis (0.5 deg) 2HG: 48%  
KES Shearing Hysteresis (5.0 deg) 2HG5: 52%  
KES Maximum Extension 500 g/cm, EM%: 142%

Relative to conventional bleaching at 50C (22/22 (50C); WI = 29), the following observations were made:

To achieve the *same degree of whiteness*, use the condition 16/16T (50C) (16mL/L H<sub>2</sub>O<sub>2</sub> (WI = 29)):

- 8% loss in specific stress\*.
- No change in elastic modulus.
- 3% increase in % strain at peak stress.
- KES Shinayakasa: 109% of CON5022
- KES Bending Hysteresis 2HB: 76%
- KES Shear Stiffness G: 85%
- KES Shearing Hysteresis (0.5 deg) 2HG: 48%
- KES Shearing Hysteresis (5.0 deg) 2HG5: 47%
- KES Maximum Extension 500 g/cm, EM%: 116%

For *better whiteness*, use the conditions of 22/8T (50C) (start at 22mL/L H<sub>2</sub>O<sub>2</sub>, but reduce to 8mL/L for Step 2 (WI = 32)):

- 6% loss in specific stress\*.
- No change in elastic modulus.
- 2% increase in % strain at peak stress.
- KES Shinayakasa: 116% of CON5022
- KES Bending Hysteresis 2HB: 76%
- KES Shear Stiffness G: 88%
- KES Shearing Hysteresis (0.5 deg) 2HG: 47%
- KES Shearing Hysteresis (5.0 deg) 2HG5: 55%
- KES Maximum Extension 500 g/cm, EM%: 120%

For *the best whiteness at 50C*, use the conditions of 22/16 (50C) (start at 22g/L H<sub>2</sub>O<sub>2</sub>, but reduce to 16g/L for Step 2 (WI = 36)):

- 4% loss in specific stress\*.
- No change in elastic modulus.
- No change in % strain at peak stress.
- KES Shinayakasa: 112% of CON5022
- KES Bending Hysteresis 2HB: 76%
- KES Shear Stiffness G: 85%
- KES Shearing Hysteresis (0.5 deg) 2HG: 43%
- KES Shearing Hysteresis (5.0 deg) 2HG5: 51%
- KES Maximum Extension 500 g/cm, EM%: 137%

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\*Relative to unbleached fabric, there is no loss in specific stress.

ARS-bleached fabrics exhibit the same or better whiteness as conventionally bleached fabrics. The added benefit is improvement in the mechanical properties related to softer fabric handle.

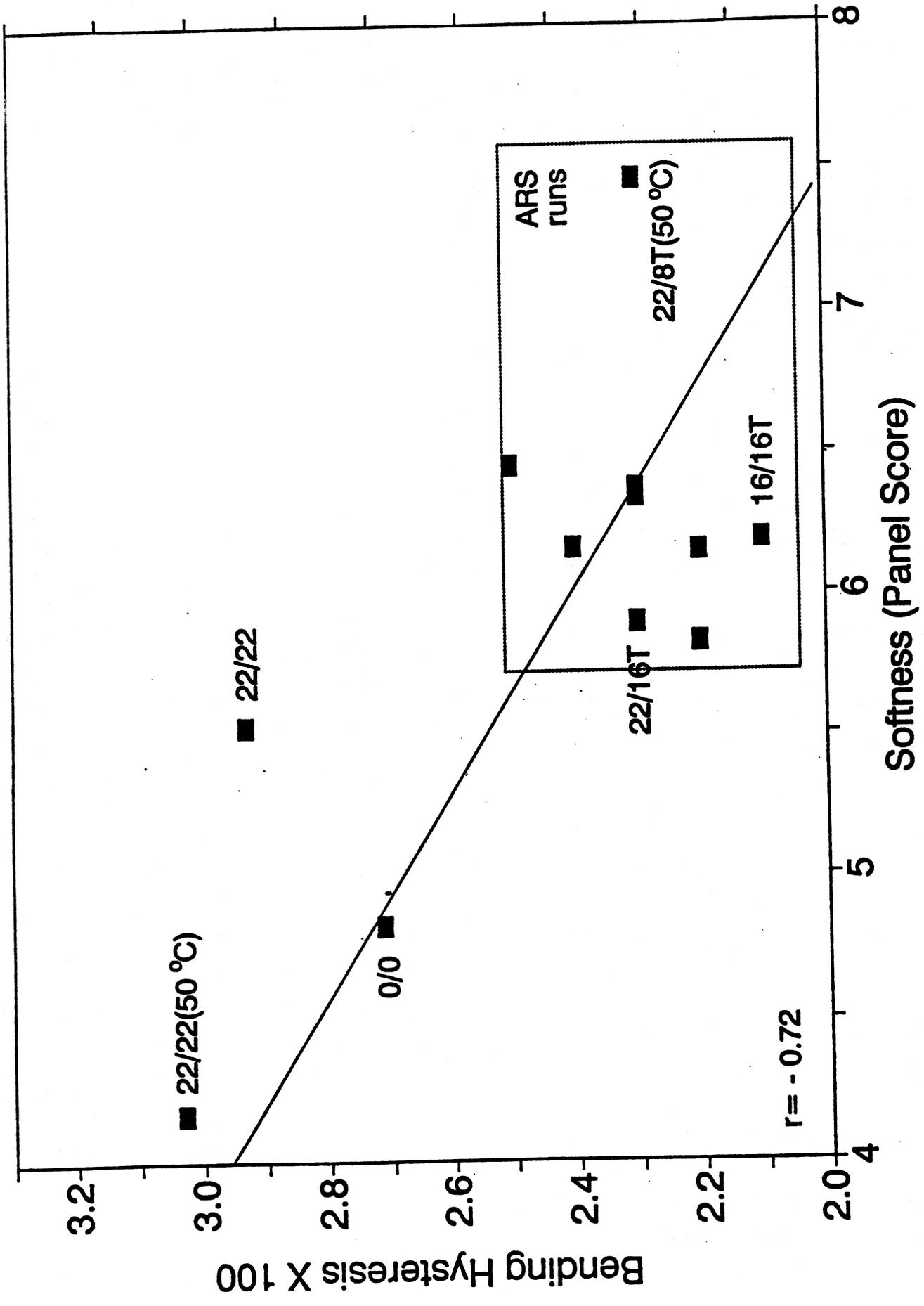
## **Conclusions**

The study done on wool challis fabric has led to the development of a standard bleaching regimen for optimum bleaching by the ARS single-bath process. That regimen (22/16T) calls for bleaching at 60C and allows for the dilution of the peroxide bath after the first step to save on the amount of required thiourea in the second step. Other conditions are also presented for improved bleaching by the ARS process, in which peroxide concentration and temperature differ from 22/16T. The optimum regimen and the other conditions all produced levels of whiteness significantly in excess of alkaline peroxide bleaching over the same time period, peroxide concentration, and temperature.

We conclude from the mechanical studies on the challis samples that strength loss from ARS bleaching is minimal relative to conventional oxidative bleaching (22/22), and for these ARS-bleached fabrics that alterations in extensibility, bending, and shearing characteristics contribute to an overall increase in softness and smooth handle.

Despite the two drawbacks to the use of the ARS process—the use of thiourea (though it is completely consumed) and the need for careful pH control, the ARS process gave us the highest achievable whiteness, permitted full bleaching in a single bath, and imparted increased fabric softness over fabrics conventionally bleached with alkaline hydrogen peroxide.

In the following paper (19), wool bleaching studies are expanded to examine the ARS process (1) on woolen flannel, especially to investigate the effects of photoexposure; and (2) on wool/cotton blended fabric and all-cotton fabric, to broaden the applicability of the ARS process to cellulosic fibers.



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Fig. 6. KES-F characteristic values for bleached worsted challis. Abbreviations: cf. Table I.

Fig. 7. KES-F hand expression, "Shinayakasa" for bleached worsted challis. Abbreviations: cf. Table I.

**Fig. 8. Shinayakasa vs. subjective score for softness for bleached worsted challis. Abbreviations: cf. Table I. Note cluster of points for ARS-bleached fabrics.**

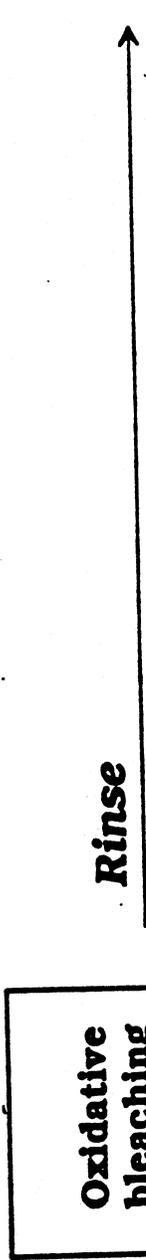
**Fig. 9. Bending hysteresis, 2HB, vs. subjective score for softness for bleached worsted challis. Abbreviations: cf. Table I. Note cluster of points for ARS-bleached fabrics.**

**Fig. 10. Specific stress and whiteness index for bleached worsted challis. Abbreviations: cf. Table I. Values with different letters are statistically different ( $p < .05$ ).**

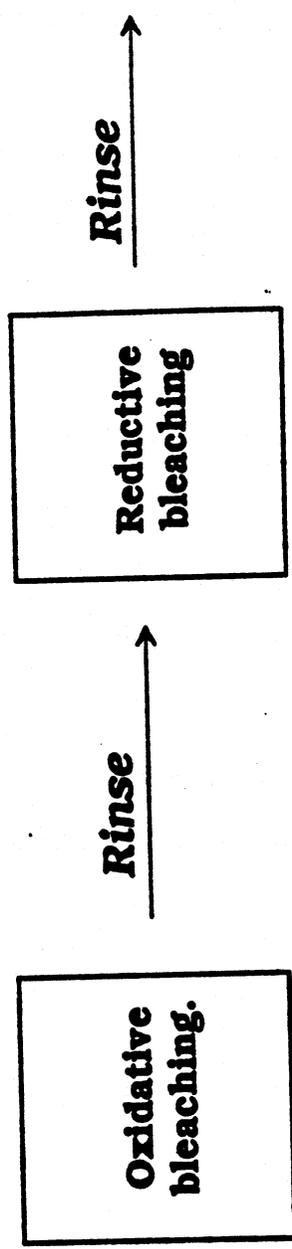
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**Fig. 12. Percent strain at peak stress and whiteness index for bleached worsted challis. Abbreviations: cf. Table I. Values with different letters are statistically different ( $p < .05$ ).**

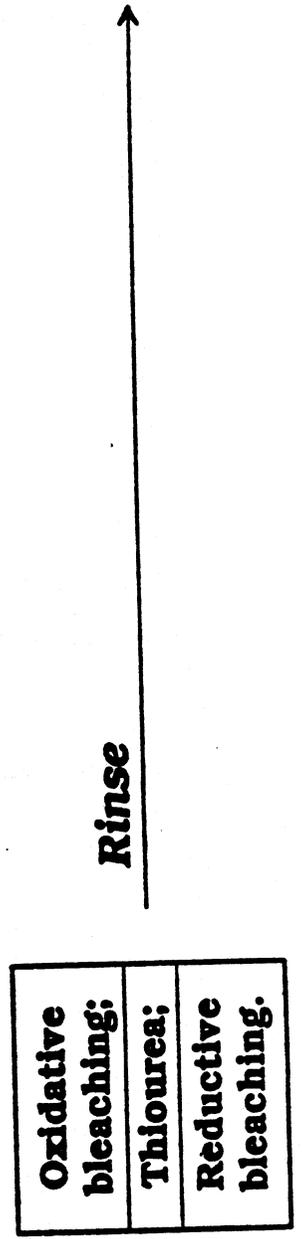
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**MOST EFFECTIVE METHOD IN PRACTICE:**

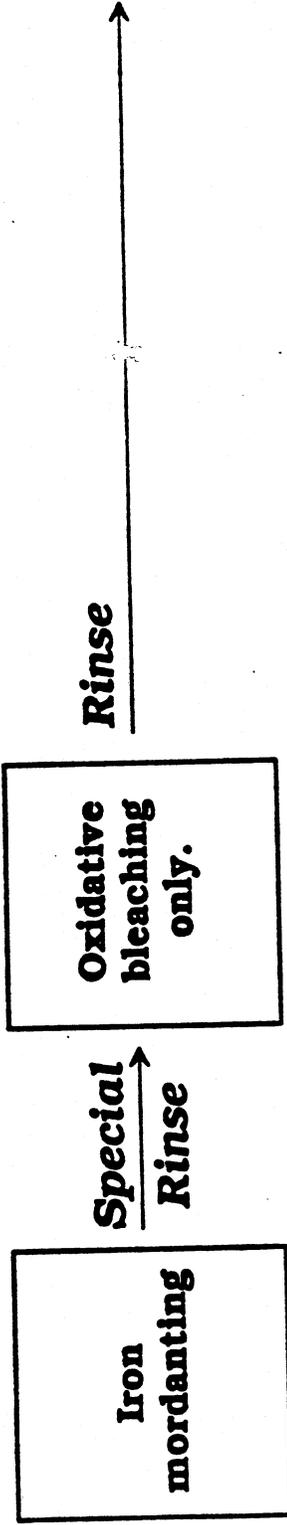


**ARS PROCESS:**

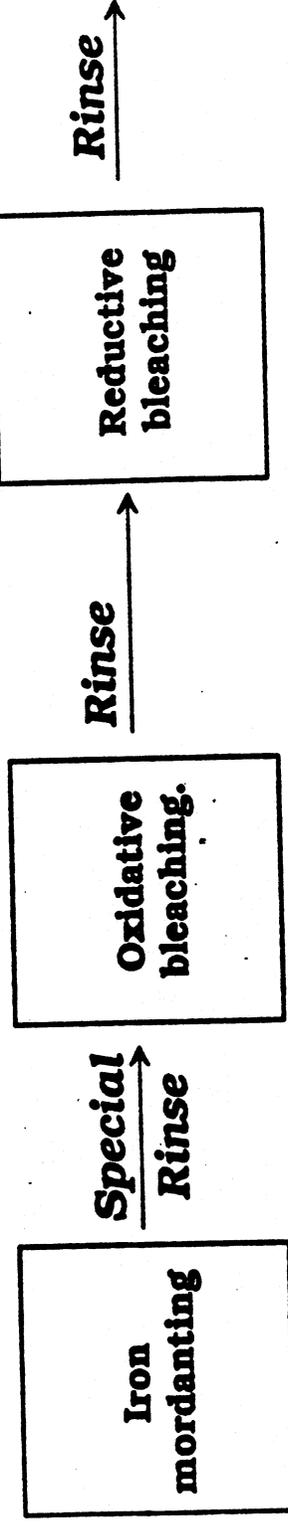


**Bleached Product**

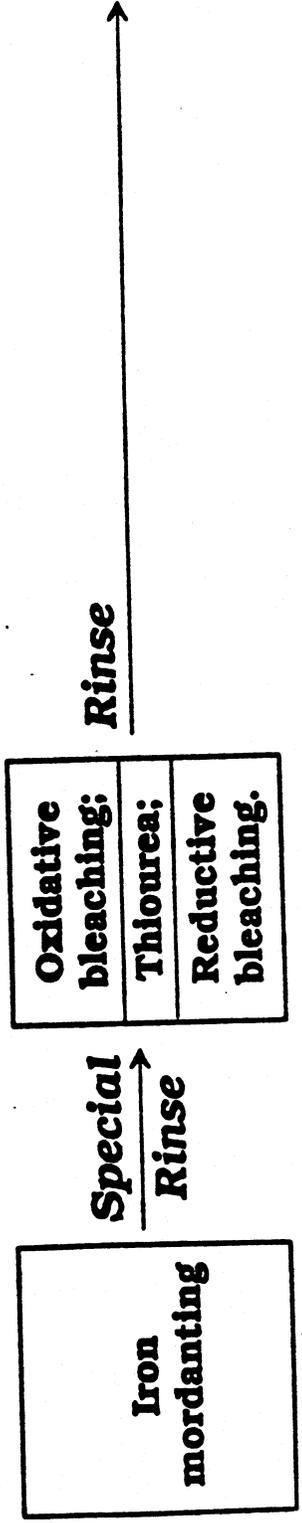
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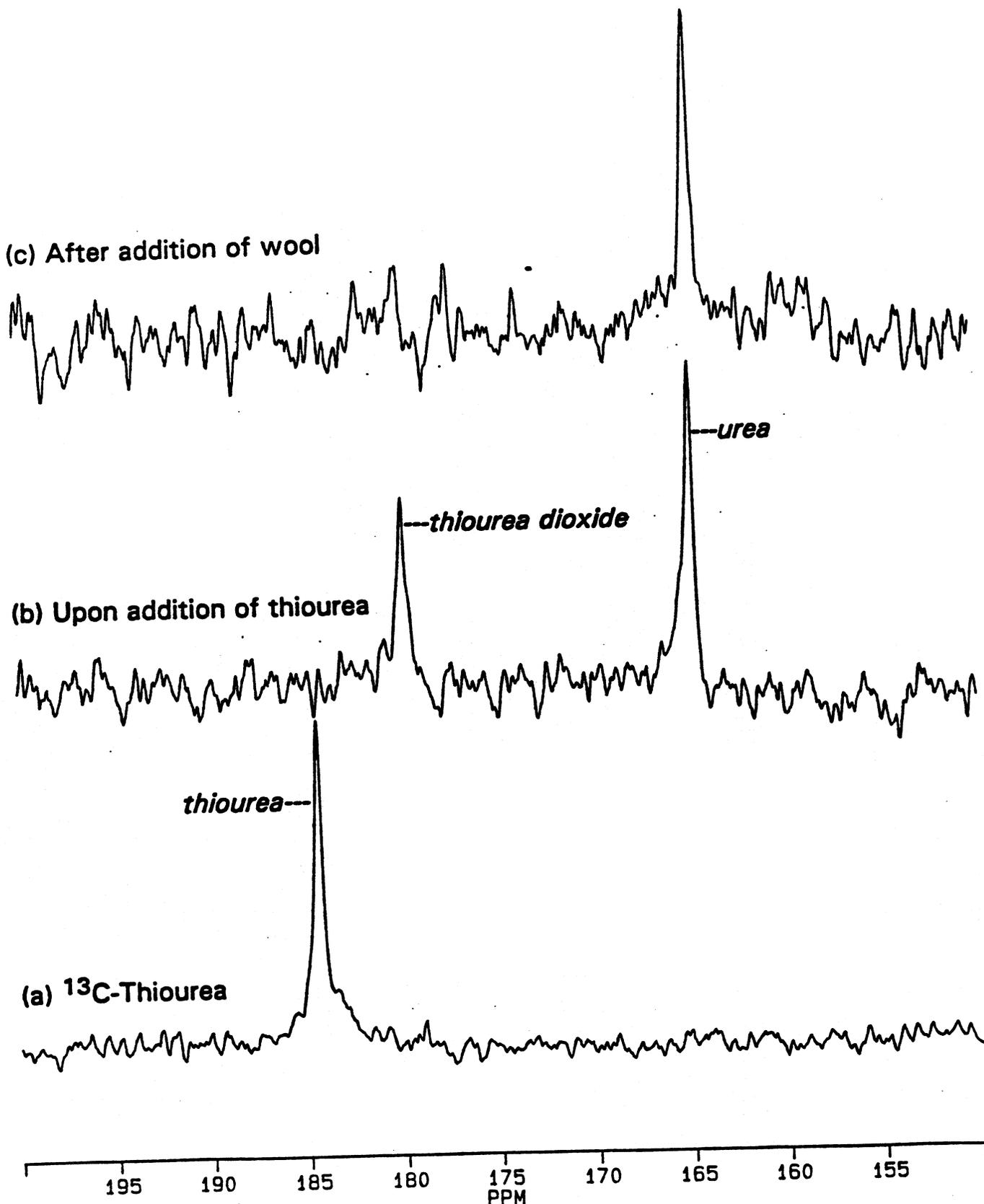
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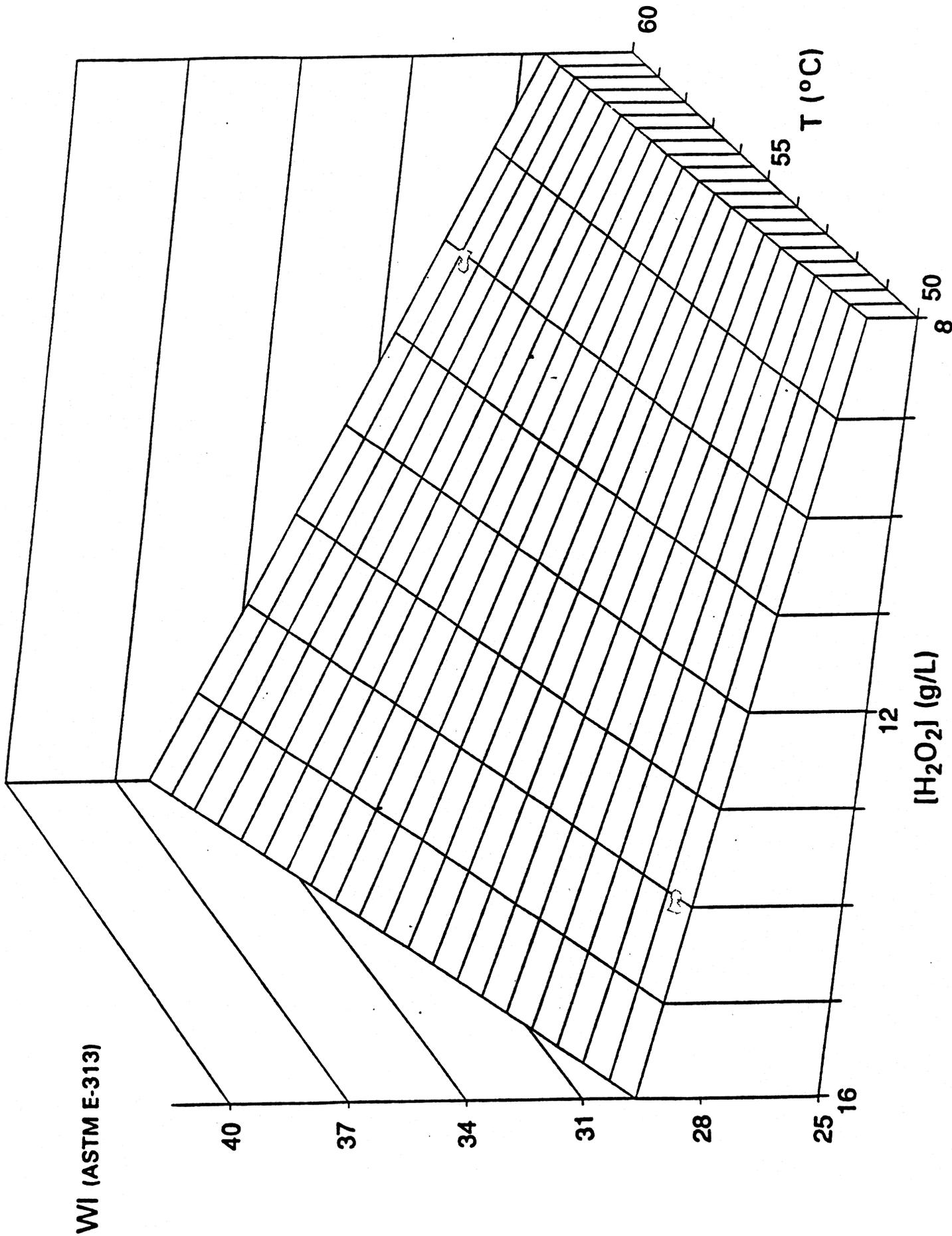


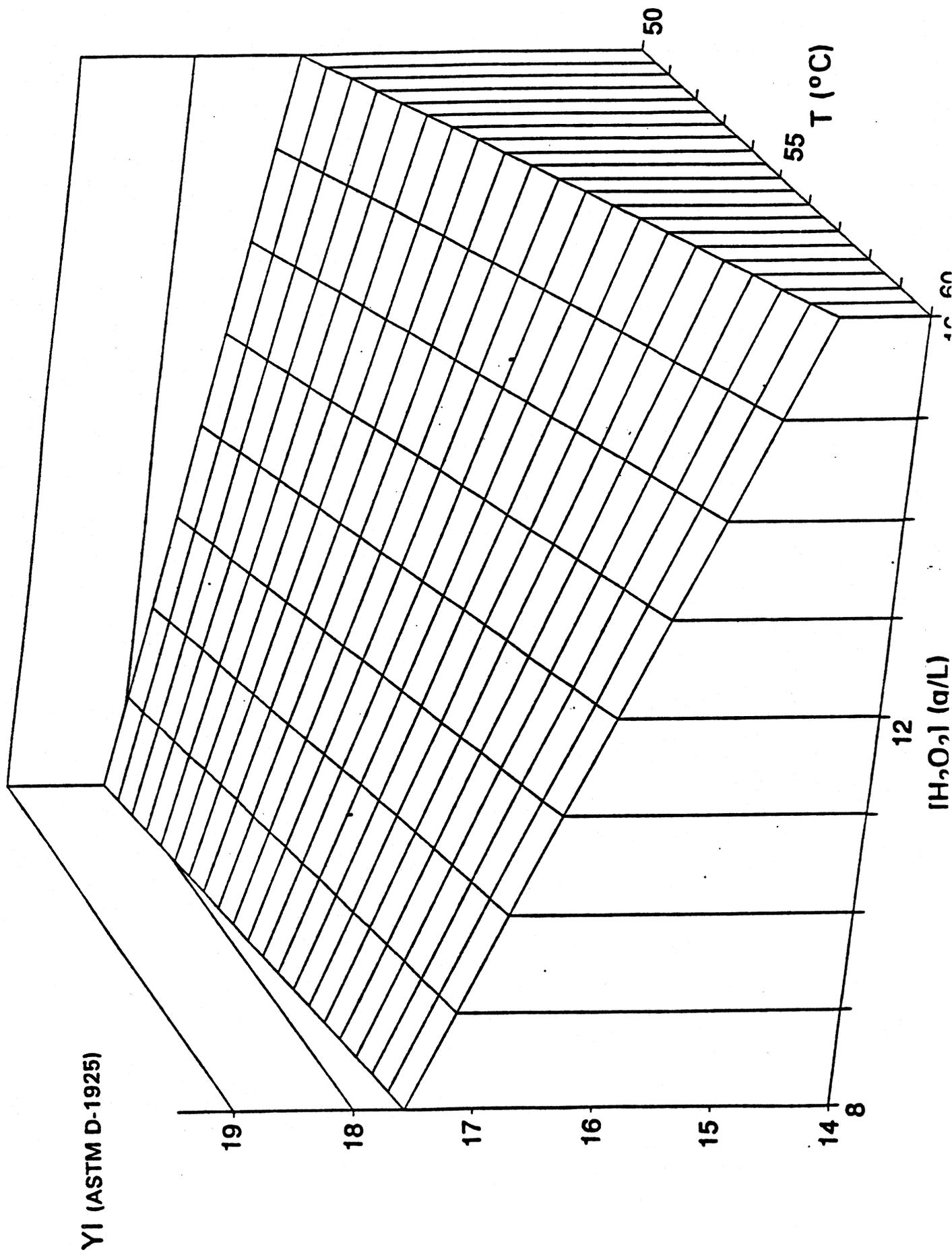
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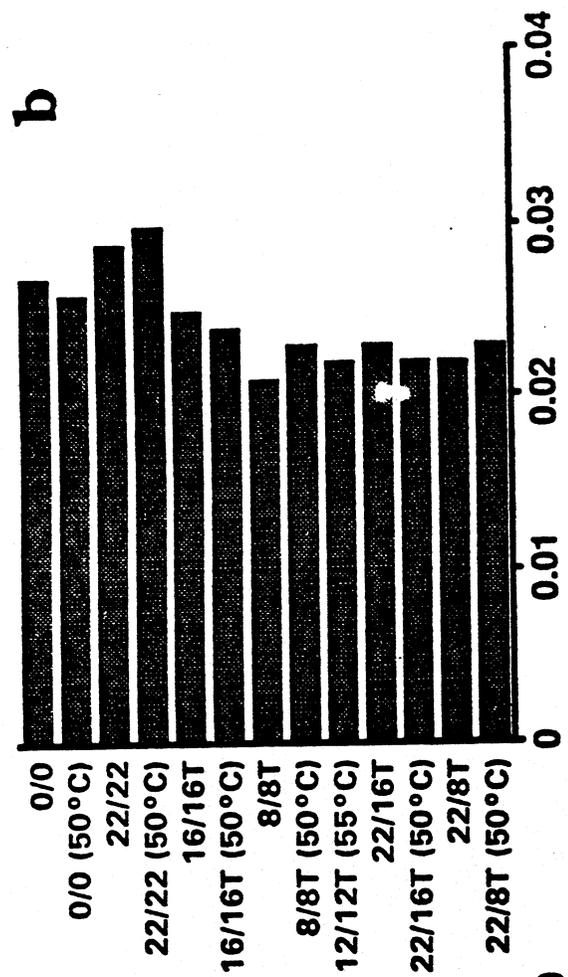
**Bleached Product**



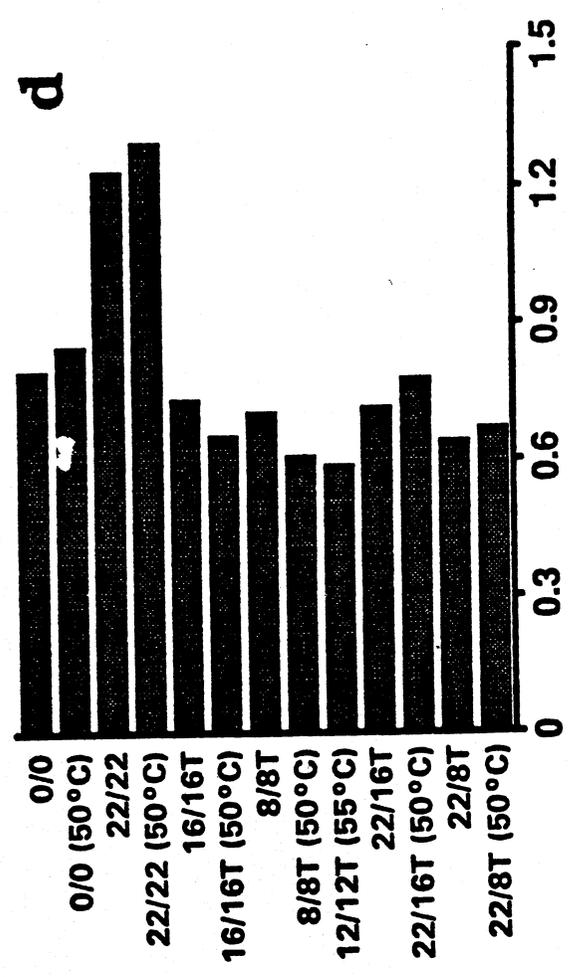




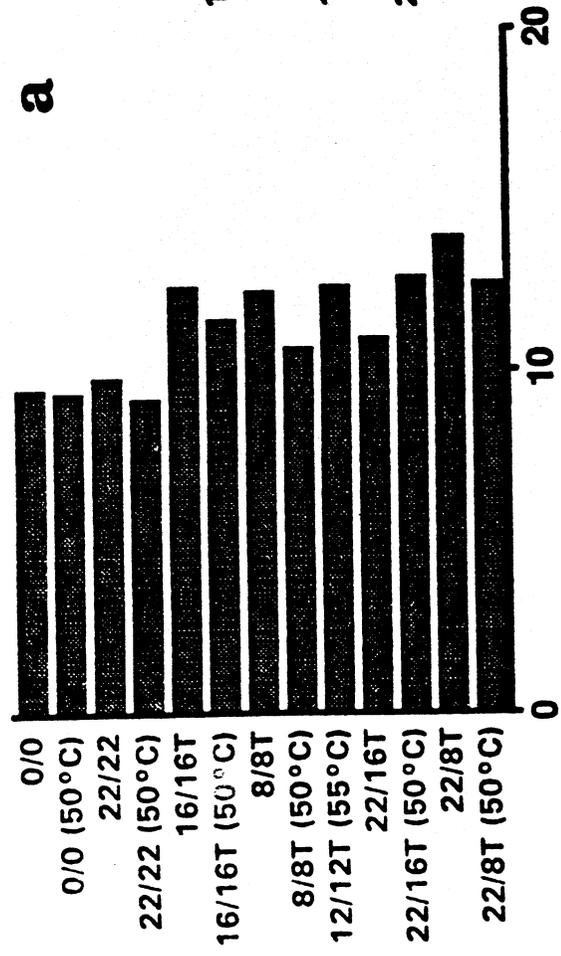
**Bending Hysteresis, 2HB**



**Shearing Hysteresis, 2HG5**



**Extension Maximum, %**



**Shearing Hysteresis, 2HG**

