

AN ABSTRACT OF THE THESIS OF

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WOOL FABRICS INCLUDING SOIL DETECTION BY X-RAY
FLUORESCENT SPECTROSCOPY

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The main goals of this study were: (1) to develop a combination of soiling, laundering, and soil detection procedures which would be accurate, reproducible, and relatively rapid; (2) to develop a quantitative method to detect artificial soil on fabrics by X-ray fluorescent spectroscopy; (3) to compare unfinished and Wurlan finished (polyamide interfacial polymerized) plain and twill weave wool fabrics for soil retention after 20 launderings; (4) to determine the effectiveness of the Wurlan finish on these woolen fabrics by measurement of shrinkage and breaking strength after laundering.

These wool fabrics were artificially soiled by immersion in a mixed oil containing 50% dibromostearic acid, dried and then tumbled with a dry kaolinite clay. Soil and shrinkage specimens were laundered with an all-purpose, hot-water anionic detergent at 120°F. for five minutes by using either gentle or no agitation.

Shrinkage specimens were also laundered with a cold-water, non-ionic detergent under the same conditions.

Count rates obtained from X-ray fluorescent spectroscopy of aluminum and bromine on soil retention specimens were converted to percentages of clay and oil respectively by comparison to fabric with known percentages of these soils. This method is faster than ashing and extraction, more accurate than color difference, less costly than neutron activation analysis, and easier than radioactive liquid scintillation.

There was a significant buildup of oil, but not of clay on these fabrics over the period of this study. Wurlan finished fabrics retained slightly more clay than the unfinished wool fabrics. This may be due to an affinity of the Wurlan finish for clay or mechanical entrapment of the clay in the fabric-finish structure. The plain weave fabrics retained similar amounts of oil, but the Wurlan finished twill weave fabric retained considerably more oil than the unfinished twill weave fabric. This difference in oil retention may also have resulted from an affinity of the finish for oil, but could reflect the felting of the unfinished twill weave fabric which made deep penetration of oil applied more difficult. There was significantly more clay and two to four times more oil retained by these fabrics when laundered without agitation than with gentle agitation.

While gentle agitation resulted in better removal of soil, it also

caused excessive shrinkage of the unfinished wool fabrics. There was significantly more warp shrinkage during the early launderings, particularly for the unfinished wool fabrics. The dimensions of the Wurlan finished fabrics were stable after ten launderings, while the unfinished fabrics continued to shrink at the 20th laundering. Wurlan finished wool fabrics shrank less than 2% in both warp and filling after 20 launderings. The shrinkage of fabrics laundered with standard anionic detergent and cold-water nonionic detergent was not significantly different.

Wurlan finish increased the warp grab breaking strength of the plain weave fabric, but did not alter that of the twill weave fabric. Limited ravelled strip breaking strength tests indicated that Wurlan finish increased the strength per warpyarn of both fabrics. Unfinished twill weave fabric laundered at gentle agitation with both detergents had a significantly higher warp grab breaking strength than the unlaundered twill weave fabric, possibly because excessive shrinkage increased the compactness of this fabric. The breaking strengths of the Wurlan finished fabrics after 20 launderings were similar to those of the unlaundered fabrics. The breaking strength and shrinkage tests seem to indicate that the Wurlan finish produced a washable wool fabric which resulted in satisfactory performance for the length of this study. These fabrics can be laundered with gentle agitation, moderately hot water, and an all-purpose, hot water synthetic detergent.

Laundering Performance of Wurlan Finished
Wool Fabrics Including Soil Detection
by X-Ray Fluorescent Spectroscopy

by

Marilyn Ruth Borchardt

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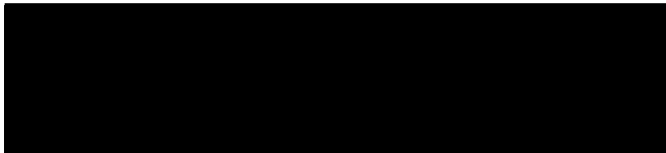
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LAUNDERING PERFORMANCE OF WURLAN FINISHED WOOL FABRICS INCLUDING SOIL DETECTION BY X-RAY FLUORESCENT SPECTROSCOPY

INTRODUCTION

Wurlan polyamide interfacial polymerization finish on wool fabrics was introduced in 1964 as "H₂O" by J. P. Stevens Co. and "Bancora" by Joseph Bancroft and Sons (Washable Wools, 1964). This finish has been recognized by textile educators as one of the most effective of the many stabilization processes which have been applied to wool fabrics (Joseph, 1966, p. 335; American Home Economics Association Textile Handbook, 1966, p. 49; Feldtman and McPhee, 1966; Potter and Corbman, 1967, p. 134, 251). In spite of this recognition of the performance of Wurlan finished wool fabrics, consumers apparently have become wary of the laundering performance of any wool fabric regardless of how it has been finished. This reluctance along with inadequate education, advertising and a limited choice of fabrics and garments has resulted in little enthusiasm or demand for such a finish.

This study is a continuation of another investigation on the soiling of wool yarns and dimensional change of Wurlan finished fabrics (Hodam, 1965). Richards (1966, p. 62-64) found some indication that Wurlan finished 65/35 mohair/wool fabric retained more oil than unfinished mohair/wool.

Statement of the Problem

The main purpose of this research was to define the performance of Wurlan finished washable woolen fabrics under home laundry conditions, using standard and cold-water synthetic detergents at 120°F. for five minutes with gentle or no agitation. This study was based on four major objectives:

1. to develop a combination of soiling, laundering and detection procedures which are reproducible, which differentiate the various laundering conditions, and which give results with a minimum of effort;
2. to perfect X-ray fluorescent spectroscopy as a quantitative method of measurement of artificial soil retained by fabrics;
3. to compare soil retention of unfinished plain and twill weave wool fabrics to soil retention of the same fabrics finished with Wurlan. using gentle or no agitation with a standard anionic detergent at 120°F. for five minutes; and
4. to compare the shrinkage and breaking strength of plain and twill weave wool fabrics, with and without Wurlan finish, after successive launderings with gentle or no agitation and standard anionic and cold-water nonionic detergents at 120°F. for five minutes.

Hypotheses

The hypotheses being tested by this study include:

1. X-ray fluorescent spectroscopy is an effective method of measurement of small amounts of soil retained by fabrics;
2. the twill weave wool fabrics retain more soil than the plain weave wool fabrics;
3. the Wurlan finished wool fabrics retain more soil than the unfinished wool fabrics;
4. fabrics laundered without agitation retain more soil than those laundered with gentle agitation;
5. the twill weave wool fabrics shrink more than the plain weave wool fabrics;
6. the unfinished wool fabrics shrink more than the Wurlan finished wool fabrics with both gentle and no agitation;
7. gentle agitation causes more shrinkage of all of the wool fabrics than no agitation;
8. nonionic detergent causes more shrinkage of all of the fabrics than anionic detergent;
9. the breaking strength of the Wurlan finished wool fabrics is greater than that of the unfinished wool fabrics prior to laundering;
10. the Wurlan finish is somewhat removed by repeated laundering, which can be detected by a decrease in breaking strength.

Delimitations

Standard synthetic detergents were used because this is the type of detergent which the consumer is most likely to have available for machine launderings. Brooks and McPhee (1967) have found that detergents are more effective than soaps in laundering of polyamide finished wool fabrics.

A temperature of 120° F. was used for laundering since previous research indicated that there was greater soil removal with little increased shrinkage of wool at this temperature rather than 70° F. (Galbraith, 1960; Hodam, 1965, p. 123).

Definitions

Built detergents are discussed in the review of literature. Both detergents used in this study contained builders.

Buffer fabric is the wool fabric added to the laundry load to give a total weight of 4.5 pounds.

Detergents are cleansing agents, including both soaps and synthetic detergents.

Clay minerals present in Spinks Bandy Black Clay include:

Kaolinite: $\text{Al}_4\text{Si}_4\text{O}_{10}(\text{OH})_8$

Mica: 1. Muscovite $\text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{OH})_2$

2. Biotite $\text{K}(\text{Mg, Fe})_3(\text{AlSi}_3\text{O}_{10})(\text{OH})_2$

Rutile: TiO_2

Anatase: TiO_2

Fabric count (yarn count, thread count) is the average number of warp ends or filling picks per square inch of fabric.

Finished refers to all specimens which have been finished by the Wurlan process (polyamide interfacial polymerization).

Plain weave is characterized by each yarn of the filling passing alternately over and under a yarn of the warp and each yarn of the warp passing alternately over and under a yarn of the filling.

S twist is a direction of twist in yarn similar to the spiral part of the letter S.

Sample is a representative portion of a lot of material which is taken for testing.

Soil checks are specimens which are soiled but not laundered at the first, tenth, and twentieth soiling to detect uniformity of soiling during the period of the study.

Specimens are a part of the sample upon which tests are performed.

Standard atmosphere for testing textiles is air maintained at a temperature of $70 \pm 2^\circ\text{F}$. with a relative humidity of $65 \pm 2\%$.

Standard detergents are defined as those most frequently used in hot water.

Synthetic detergents are cleansing agents made from petroleum

rather than natural fat.

Twill weave is characterized by distinct diagonal lines produced by warp yarns passing over one or more filling yarns and then under two or more filling yarns or vice-versa with progression of interlacing for one or more to the right or left.

Unfinished refers to all specimens to which Wurlan finish was not applied.

Yarn twist is the number of turns about its axis per unit of length of yarn.

REVIEW OF LITERATURE

Literature related to this study includes investigations of and theories concerning: soiling, detergency, shrinkage and breaking strength of wool fabrics, and recommendations for machine laundering of wool fabrics.

Soiling and Soil Removal

From the homemaker's viewpoint, soil may be defined as matter out of place. Soil picked up by fabrics may be generally classified as oily or particulate (J. C. Harris, 1958), and varies in complexity and composition depending upon the physical environment and occupation of the wearer.

Composition of Natural Soils

Particles present in the atmosphere include dusts, fumes, smokes, mists and fogs. Dusts are formed by disintegration processes like grinding while fumes result from combustion, sublimation, or distillation. This dust contains particles which are usually less than one micron in diameter and have active Brownian motion. Smokes are organic compounds, often associated with oil and grease, while mists and fogs are produced from the disintegration of a liquid or condensation of vapor (Friedlander et al., 1952, p. 1).

An analysis of samples of vacuum cleaner dust obtained from widely separated geographical locations and from different homes and commercial establishments revealed a similarity in chemical composition with:

11.4-15.9% Water soluble material

4.9-12.8 Ether soluble material

0- 3.8 Moisture

24.0-28.9 Carbon

50.5-57.8 Ash

0.5- 0.8 Carbon black equivalent

The particle sizes of this natural soil from six cities indicated that 53% of the particles were less than four microns in diameter, whereas, only 17% of the particles were larger than 20 microns (Sanders and Lambert, 1950, p. 155). Vacuum cleaner dust from an office in Germany was similar in content and particle size to that found by Sanders and Lambert (Goette, 1960).

One of the main constituents of natural soil is clay, either carried by air or suspended in water, with kaolinite and montmorillonite clays present in abundance (Schott, 1965b). Clay particles are readily picked up by fabrics and are among the most difficult inorganic components of natural soil to remove from fabrics (Getschell, 1955; Schott, 1966). Powe (1959) found that clay minerals that varied in diameter from 20 millimicrons to one micron were the main inorganic

components retained by naturally soiled cotton clothing.

Analyses of fatty matter in natural soils indicated that air-borne soil contained 22% oil; city dirt from seven cities contained 4.9-12.0% oil; carpet dirt contained 6.0% oil; and domestic garments contained from 1.0-27.0% oil (J. C. Harris, 1958). An analysis by Wagg and Britt (1962) of articles sent to the laundry indicated that as much as 80% of the soil on the fabrics was fatty excretion of the sebaceous glands of the skin, indicating that such fabrics have a high ratio of oil to inorganic components. This difference in oil content could be accounted for by the fact that bedding and shirts are the most common articles commercially laundered, whereas Harris analyzed a variety of clothing articles.

The composition of the oily portion of natural soil on shirts has been identified by Brown (1947) as 31.4% long chain fatty acids, 29.2% long chain neutral fats (triglycerides), 3.3% short chain fatty materials, 15.3% acetylatable materials such as fatty alcohols and cholesterol, and 21.0% hydrocarbons. The total weight of oily materials on collars of shirts was found to be about 1% of the weight of the fabrics. In a more recent analysis of oil content on naturally soiled cotton garments, Powe and Marple (1960) found 15-30% fatty acids, 12-16% wax esters, 30-50% triglycerides, 10% mono- and di-glycerides, 3% cholesteryl esters, 3% cholesterol, 10-12% squalene and 1-3% hydrocarbons. Of these components, the long

chain fatty acids were the most difficult to remove when single component soil systems were compared. However, all components of mixed oils were removed from cotton equally, suggesting that artificial soils probably should contain mixtures of oils to obtain realistic results.

Artificial Soils

Analyses of natural soils brought about an increasing awareness that artificial soils being used were not very realistic and, therefore, might not correlate with actual performance. The artificial soils used most frequently by early researchers included carbon compounds, iron oxide, ilmenite, zirconyl phosphate, barium titanate and carpet soil (Davis, 1963). Carbon black and graphite soils have been favored by many researchers in the past, but are not used very often today because neither material is present in large amounts in naturally soiled clothes (Martin and Davis, 1960). A variety of vegetable oils have also been used as substitutes for natural oils. A comprehensive discussion of carbon black soils, as well as particulate and oil mixtures used in the past was given by Hodam (1965, p. 19-22, 27-32, 146-149).

Recent studies have used clays as a component of artificial soils since analyses of natural soils have indicated that the inorganic component of soil causing the greatest soiling problem was clay

(Sanders and Lambert, 1950; Martin and Davis, 1960; Tuzson and Short, 1962; and Short, 1965). The clays used most frequently as components of artificial soils have been calcium and sodium montmorillonite or kaolinite. Davis (1963) used Spinks Bandy Black Research Clay (about 80% kaolinite) to soil cotton and found an excellent correlation between the clay content and the reflectance which resulted. Comparison of the reflectance of cotton swatches soiled by clay suspensions of various concentrations with swatches of laundered naturally soiled articles resulted in correlations to within one reflectance unit.

Recently clays and oils have been combined to give an artificial soil containing the components which are most difficult to remove from naturally soiled fabrics. It is recognized that other types of soil may contribute to natural soiling of garments, but it is considered impossible and unnecessary to exactly duplicate natural soil. Studies using artificial soil can predict the soil removal from naturally soiled fabrics under given conditions, but they cannot be assumed to give the same results and, therefore, must be substantiated by actual use studies.

Mixed soils used recently include (1) a combination of oleic acid and Spinks Bandy Black Clay (Davis, 1963; Kennedy and Stout, 1968), (2) a combination of calcium montmorillonite clay labeled with ^{59}Fe and a mixed oil containing 33% palmitic acid (0.78%

labeled with ^{14}C), 32% olive oil, 20% squalene and 15% n-decyl alcohol (Hodam, 1965, p. 64), (3) a combination of Spinks Bandy Black Clay and a mixed oil containing 7% cetyl alcohol, 25% triolein, 15% stearic acid, 15% oleic acid, 5% cholesterol, 8% squalene, and 25% tristearin (Oregon. State University, 1968), (4) a mixture of 75% Spinks Bandy Black Clay with 25% fatty acids, esters, alcohols and hydrocarbons (Tomlinson, Billica and Sloan, 1966), and (5) a combination of montmorillonite (bentonite) and a mixed oil containing 20.0% dibromostearic acid, 5.6% cetyl alcohol, 20.0% triolein, 12.0% stearic acid, 12.0% oleic acid, 4.0% cholesterol, 6.4% squalene, and 20% tristearin (Richards, 1966, p. 32, 34).

Soiling of Fabrics

Generally garments are soiled by the atmosphere or by contact with soiled objects and the skin. The mechanisms which bring soil particles into contact with fibers include: diffusion from higher concentrations to lower concentrations for particles smaller than 0.5 microns; impact with the clothing and inertial effects of rapidly moving air for particles larger than about one micron; direct transfer or contact with liquid droplets on the clothing; and electrostatic attraction. These electrostatic forces can be created by friction or violent collisions with other particles (Friedlander et al., 1952, p. 4-5, 12; AATCC New York Section, 1952).

After initial contact between soil and fibers, adherence can occur by mechanical forces such as occlusion in pits and crevices on fiber surfaces, by 'oil' bonding, and possibly by electrical forces (AATCC New York Section, 1952). Van der Waals attraction may also exist between soil and fabric over very small distances, and some chemical bonds similar to dye or finish interaction with fabric are possible in natural soiling and may explain the difficulty of removal of some soils (Trowbridge, 1964).

The soiling of a particular fabric depends upon its history, as well as the soil with which it comes in contact. For example, fabrics heated or predried before soiling, retain soil much more tenaciously than fabrics that have an adsorbed water layer that can hold soil at a distance from the fiber surface (J. C. Harris, 1958).

The presence of oil on fabrics appears to influence retention of particulate matter by some fabrics. Several researchers have confirmed the observation that oily material or detergent residue left on fibers causes rapid resoiling by particulate matter. Leonard (1950) found that wool fibers soiled more readily in actual use when some oil or detergent was present. Weatherburn and Bayley (1955) also noted that the presence of even small amounts of oily material in yarns increased the amount of vacuum cleaner soil retained by wool and viscose rayon. The American Association of Textile Chemists and Colorists, New York Section (1952) found that during

tumbling, wool fabrics picked up larger amounts of bone charcoal, powdered sodium sulfate and natural rug soil as the amount of oil present on the fabric increased. Tomlinson, Billica and Sloan (1966) have studied the natural soiling and redeposition of cotton polyester fabrics. They concluded that:

If deposition of fatty materials could be prevented or if a wash process could be devised which would completely remove the oily constituent of natural dirt, the particulate constituent might give little trouble (p. 12).

However, Howarth and Piper (1966) replied that loosely held particulate soil presented few problems even if it was attached to a fatty film. They maintained that this attachment to oil sometimes may even assist in soil removal. It was pointed out that launderers, dry cleaners, and textile finishers prefer to have particulate soil attached to a fatty film rather than firmly attached to the textile itself.

In an early study of soiling of fabrics of various fiber contents, Masland (1939) concluded that the primary controlling factors in soil retention were the cross-sectional outline and diameter of the fiber. In support of this research, J. C. Harris (1958) has noted that cotton and wool soil more readily than synthetics and soil is more difficult to remove from wool fabrics. An electron microscopic investigation by Kling and Mahl (1954) demonstrated that particulate soil concentrated on the edges of wool scales indicating that this structural feature of wool fibers might be expected to cause greater retention of

soil than a smooth fiber contour.

Research with a variety of synthetic fibers has indicated that some factor other than fiber diameter and cross sectional outline is important in affinity for soil. The American Association of Textile Chemists and Colorists, New York Section (1952) found that fabrics of wool with rough cross sectional outline and nylon with a smooth outline adsorbed more of both vacuum cleaner dirt and a mixed synthetic soil modeled after vacuum cleaner soil (both applied by four different methods) than "Dacron" polyester, "Orlon" acrylic, rayon, acetate, silk or cotton fabrics. Weatherburn and Bayley (1957a) also studied the soiling of acetate, nylon, rayon and "Terylene" polyester with dry vacuum cleaner soil. They found that there were differences in soil retained by fibers of various chemical types that could not be related to differences in physical shape and size of yarns. The soiling of synthetic fibers in spite of smooth cross sectional outline has been elucidated more recently by Davis (1966). He found that synthetic fibers that were initially quite smooth, developed surface abrasions during wear which acted very much like the natural rugosities of cotton and wool. Clay minerals in particular were extremely difficult to remove from fabrics with abraded surfaces. This study helps to explain why previous workers did not find a clear cut difference in the soiling properties of natural and synthetic fibers.

The fabric geometry and finish also influence both the ease of soiling of fabrics and the removal of soil. Several investigations of the influence of yarn structure have indicated that spun yarns soil more readily than filament yarns (AATCC New York Section 1955; J. C. Harris, 1958). The effect of twist on filament acetate yarns soiled with vacuum cleaner soil by dry tumbling was studied by Weatherburn and Bayley (1957b). As twist was increased, soil retention increased rapidly to a maximum occurring at less than 20 turns per inch and then decreased to values below that of the untwisted yarn. Microscopic examination indicated that 20 turns per inch allowed visible soil particles to penetrate to the center of the yarn while at 60 turns per inch the soil was located only on the exterior of the yarn.

Soiling of cotton, wool, silk, nylon, and linen with lampblack by Hart and Compton (1953) indicated that inter-fiber and inter-yarn entrapment of soil particles was the major factor in retention during soiling and after drying of the soiled fabric. They concluded that, generally as the weight and complexity of the fabric structure increased, the number of macro-occluded soil particles increased. Tripp et al. (1958) similarly found that both natural soiling of carpets and artificial soiling with carbon black, ceramic clay, mixed synthetic soil and carbon black were influenced by the yarn and fabric geometry. They also noted that large amounts of soil were

attached to fiber surfaces in areas free from irregularities.

Finishes may alter the soilability of a particular fiber-fabric system. In a study of finishes that impart dry soil resistance to cotton, Porter et al. (1957) found that both natural carpet soiling and artificial dry soiling with carbon black, ceramic clay, charcoal, and mixed synthetic soil were reduced on the finished fabrics simply because the finish sealed off inter-fiber spaces in yarns, thus reducing the total surface area.

Removal of Clay and Oil from Fabrics

In a comparison of kaolinite and sodium montmorillonite remaining on cotton after laundering, Schott (1965a, 1965b) found that a larger amount by weight of kaolinite was retained. However, due to the smaller particle size of montmorillonite, a larger number of these particles were retained. He concluded that the kaolinite was gradually removed mechanically because the larger particles are stiffer and are not able to make such firm contact with the fabric. In another study, Schott (1966) compared the removal of sodium and calcium montmorillonite and found that the sodium montmorillonite was retained by the cotton fabric, whereas the calcium montmorillonite was gradually washed off by water. It is known that calcium montmorillonite flocculates readily in water resulting in a larger effective particle size comparable to kaolinite (Van Olphen, 1963,

p. 24).

Short (1965) concluded that there were at least three mechanisms of removal of kaolinite clay (particle size of four to five microns in diameter) from cotton. During the initial period of the removal process, occluded particles were removed by combined mechanical action and bulk fluid flow. For the next 180-240 seconds there was migration of particles from the fabric surface through the boundary layer of the fluid to the bulk solution in a rate process. The process for the remainder of the cleaning period was a combined chemical-physical bonding in which the addition of chemical and thermal energy were required.

In a study of the removal of montmorillonite clay and mixed oils from Wurlan finished wool fabrics, Richards and Morris (1968) concluded that vigorous agitation was necessary for complete removal. Recent work by Oldenroth at Krefeld Institute in Germany (cited in Davis, 1966) compared the removal of natural soil from cotton, nylon and polyester undershirts, with the cotton undershirts laundered at 203°F. and the synthetic fiber garments at 140°F. After the tenth laundering, three to four times more organic soil was extracted from the cotton garments than from the synthetic fiber garments indicating that under these conditions, the cotton retained more body oil than polyester or nylon.

Studies of the removal of natural soil from cotton (AATCC

Washington Section, 1954) indicated that the major portion was removed during the first minutes of laundering, and the rate of removal after four minutes was negligible. Davis (1963) similarly found that the greatest amount of oleic acid and Spinks Bandy Black Clay was removed from cotton during the first five minutes of laundering. A radioactive tracer study by Wagg and Britt (1962) also indicated no further significant removal of radioactive mixed oils and graphite from cotton, wool, rayon, nylon, polyester and acetate after three minutes of laundering. Kennedy and Stout (1968) demonstrated that successive laundering of fabrics of various fiber contents resulted in some further removal of artificially applied oleic acid and Spinks Bandy Black Clay, indicating that a new wash bath is necessary for further significant removal of some soils.

Very little research has been done to determine whether the removal of particulate soil is influenced by the presence of oil. In one instance Ultermohlen (1949) found that removal of lampblack and iron oxide from cotton was not dependent upon the presence or absence of oil. Hodam (1965, p. 121) also noted no relationship between montmorillonite clay and palmitic acid retained by wool fibers after laundering.

Redeposition of soils on fabrics has been investigated by several researchers. Powe (1963) found that under laundry conditions with temperature below the melting point of oils, reduced agitation, and

less than two minutes of wash, sebum accumulated on cotton and increased the redeposition of particulate soil because the oily fibers adsorbed more soil from the wash water than clean fibers. More recently Tomlinson, Billica and Sloan (1966) observed similar redeposition on unworn polyester fabrics laundered 20 times in a commercial laundry. A continuous layer of redeposited oily soil surrounded each filament and concentrated near fiber-fiber junction points with the particulate soil embedded in the oily soil layer. In another study, Powe (1959) used an electron microscope to observe a layer of amorphous material in which a number of particles were imbedded on an old laundered cotton undershirt. There is some indication that the type of finishes may influence the reaction of the fabric to redeposition of soil. Berch and Peper (1963) found that the presence of amino groups in a finish applied to cotton fabrics caused heavy redeposition with iron oxide, carbon black and vacuum cleaner suspensions.

Artificially Soiled Fabrics

Artificially soiled fabrics vary greatly depending upon the objectives of the study:

It is apparent from the literature that the effect of the many variables, such as fiber, fabric construction, finishing treatments, and the different types and particle sizes of soils, makes it extremely improbable that any single test will ever predict the soiling characteristics

of a fabric, but rather that the integrated information of several different tests will be necessary to characterize this property of a fiber or fabric (AATCC New York Section, 1955, p. 816).

Standards of performance of artificially soiled fabrics have been recognized to insure that these tests be as realistic as possible.

Important requirements for an artificially soiled fabric include:

1. uniform and representative soil that is easy to obtain,
2. uniform soil application with a minimum of aging effects,
3. reproducible results at different applications, as well as within a single application,
4. reproducible ranking of detergents in the same order as obtained under practical conditions, and
5. sufficient sensitivity to distinguish known differences between detergents with a minimum expenditure of time and effort (J. C. Harris, 1954, p. 65-66; Diehl and Crowe, 1955).

There have been many attempts to formulate a single artificial application of soil to fabrics suitable for diverse conditions of soiling and soil removal. However, a variety of methods are still in use today. Methods of application of soil to fabrics have included: light abrasion of soil onto fabrics using an abrasion machine, ball mill, blower, tumbling shaker, or a vapor chamber. Liquid soils have been applied by smearing, immersing, padding, or spraying with a soil suspension (AATCC New York Section, 1955; Iig, 1967).

The majority of current artificial applications use immersion,

dry or paste abrasion, or dry tumbling to apply soil to fabrics. In one immersion method, fabric was tumbled with a mixture of Spinks Bandy Black Clay and oleic acid in perchlorethylene in a Launder-Ometer (Kennedy and Stout, 1968). Another immersion technique involved the application of mixed oil and clay separately. The oil was dissolved by perchlorethylene and the fabric immersed in this solution. After drying, the fabric was tumbled in the Launder-Ometer with a clay-water suspension (Richards and Morris, 1968; Oregon State University, 1968).

The immersion method of soil application was rejected by Sanders and Lambert (1950) because they felt it was not realistic since the process is similar to redeposition in which the finest particles penetrate deeply into the fabric, resulting in stains. They experimented with a Schiefer abrasion machine to apply a combination of oil and dry components as a dry soil. It was difficult to apply the same amount of soil to replicates, but they justified this variation by pointing out that at least 95% of the soil applied was removed in the laundering process anyway.

Several workers have investigated the dry tumbling method of soil application in detail. The American Association of Textile Chemists and Colorists, New York Section (1952) applied a filtered vacuum cleaner soil to a variety of fabrics. X-ray fluorescent spectroscopy indicated that the elements of the soil were applied in the same ratio

occurring in the original soil. They concluded that the dry process did not add preferentially any particular elements of this complex soil. One of the most recent methods of application of dry soil by tumbling was developed by Berch et al. (1967). This method involved soiling of dense felt cubes with a known amount of any type of particulate or oily soil by tumbling, followed by tumbling the soiled cubes with the fabric. This method resulted in a high degree of soiling uniformity on single cotton specimens, but variation between specimens was eliminated only by soiling a large group of specimens in the same jar at one time. The main difficulty with both dry tumbling and liquid immersion applications is uniform and reproducible deposition to fabrics and control of the amount of soil applied (Schwartz et al., 1952; Tomijama and Jimori, 1965; Nettelstroth and Viertil, 1965; E. I. DuPont de Nemours, n.d.).

Measurement of Soil Retention

The measurement of soil retention can be either an absolute or a comparative measure of soil in the wash water or on the fabrics (J. C. Harris, 1954, p. 8). Absolute methods most frequently used include gravimetric procedures and radioactive tracers to determine the amount of soil on the fabric or in the wash liquor, and X-ray fluorescent spectroscopy to analyze soil on fabrics. Comparative methods used include measurement of reflectance

of fabrics or measurement of turbidity of the wash liquor.

The amount of soil on fabrics before and after washing has been determined by extraction and ashing to obtain oil and mineral content respectively. While this is more indicative of the amount of soil removed during laundering than methods such as reflectance, other methods are preferred because they are faster. It has been recognized that the weight change during extraction and ashing may include detergent residues and other contaminants (Richards, 1966, p. 68-69). Ashing and extraction have been used along with reflectance measurements by the Oregon State University, Department of Home Economics Research (1968). In an investigation of the feasibility of using X-ray fluorescent spectroscopy to detect soil on fabrics, Richards, Morris and Arkley (1968) compared these results with extraction and ashing data. The amount of soil in the wash liquor has also been measured by gravimetric procedures (Schoenberg, 1961).

Radioactive tracers have been used to estimate minute amounts of soil, to calibrate other methods, and to determine the type and quantity of soil (J. C. Harris, 1954, p. 8). Wagg and Britt (1962) used radioactive tracers in a complex synthetic oil mixture to determine the rate of removal from chopped cotton, wool, nylon, acetate, polyester and rayon. Hodam (1965, p. 77) used a radioactive tracer to determine the soil removal from wool yarns by liquid scintillation counting of both yarn and wash liquor. The main

limitation of using a radioactive tracer is the necessity to add a radioactive portion which may differ in crystal shape, particle size, and other characteristics, and, therefore, be removed in a different manner than other components of the soil (Schoenberg, 1961).

X-ray fluorescent spectroscopy is one of the most recent techniques used to determine the amount of soil retained by fabrics. This technique was used previously to determine the elements present in modified cotton fabrics, and it was found that heavy elements could be quantitatively detected at levels as low as 0.01% (Tripp et al., 1964). The American Association of Textile Chemists and Colorists, New York Section (1952) utilized X-ray fluorescent spectroscopy to determine if all elements of a dry filtered vacuum cleaner soil were attached to fabrics of various fiber contents in equal amounts by the dry tumbling method. More recently Richards, Morris and Arkley (1968) used X-ray fluorescent spectroscopy to indicate the relative amounts of clay and oil present on Wurlan finished wool fabrics after laundering.

Reflectance has been used as a comparative measure of residual soil by a number of researchers (Sanders and Lambert, 1950; Bacon and Smith, 1954; AATCC New York Section, 1955; Martin and Davis, 1960; Berch et al., 1967; Kennedy and Stout, 1968; Oregon. State University, 1968). Reflectance indicates the

improvement in visual cleanliness of fabrics (Schoenberg, 1961). Wagg (1953, 1957) has pointed out that reflectance measures the removal of colored components of the soil, but does not necessarily detect oil, necessitating the use of at least one other method of detection such as extraction. A comprehensive discussion of the limitations of reflectance to detect soil retention is given by Hodam (1965, p. 7-12).

The amount of soil in wash liquor has been measured by light transmission (turbidity) methods by Vaughn and Suter (1950) and Bradacs and Schletelig (1964). Baker and Kern (1950) pointed out that turbidity methods often give erroneous results for measurement of soil removal.

Detergents and Detergency

Detergents

In recent years there have been a number of significant changes in home laundering surfactants. Perhaps the most important of these is the almost universal replacement of soap by synthetic detergent. Today only about 5% of all household cleaning products are soaps (Strawn, 1966, p. 4). Since many of these soaps are not suitable for machine laundering, some homemakers may use standard synthetic detergents or cold-water detergents to machine

launder washable woolens. For many years authorities have recommended the use of unbuilt soaps for laundering wool fabrics, but recently a number of studies have indicated that synthetic detergents may also be used (Furry and McLendon, 1950; Galbraith et al., 1955; Galbraith, 1960; Furry, 1963, p. 6; Hodam, 1965, p. 121-122). A study by McPhee (1961a) indicated that almost any soap or synthetic detergent, except strong alkali soap in hot water, could be used to launder wool. Flett (1946) found that the laundering of wool with synthetic detergent, alkyl aryl sulfonate for two minutes gave a whiteness comparable to the use of soap for 20 minutes.

The two main types of synthetic detergents used for home laundering today are anionic and nonionic, with the anionic being more commonly used. Most anionic detergents contain alkyl aryl sulfonate, which is similar in structure to soap except that the hydrophobic end is derived from a petroleum fraction rather than from a fat (Price, 1952, p. 45). Most of these anionic detergents are marketed as a powder (Cohen and Linton, 1961, p. 53). Anionic detergents act by formation of an anion (negative charge) at the hydrophilic part of the detergent molecule when dissolved in water, and are most effective in removal of soil which is strongly ionic, such as clays and other minerals. The anionic agent appears to be adsorbed on both the fabric and soil surfaces decreasing the attraction between the charged surfaces by neutralizing positively charged centers or

increasing the degree of negative charge on both the fabric and the soil (Trowbridge, 1964). However, anionic detergents such as alkyl aryl sulfonate have poor suspending and emulsification properties, and additives such as carboxy methyl cellulose, sodium sulfate, sodium metasilicate, or trisodium phosphate are often used to aid in suspending dirt (Speel, 1952, p. 305).

Nonionic detergents are composed of hydrophilic and hydrophobic portions, but do not dissociate in water. Nonionics often contain 100% active ingredients, may be either liquid or powder, frequently are of greater density than other detergents, and have a lower foaming action (Cohen and Linton, 1961). According to Trowbridge (1964) nonionics are generally more effective in removal of oily soils due to reduction of van der Waals forces when the surfactant is adsorbed onto the hydrophobic oil surfaces. The water soluble portion of the detergent prevents the redeposition of the oil.

Since 1965, all detergents have been biodegradable, containing a straight chain alkyl aryl sulfonate which is easier for microorganisms to break down than the more highly branched alkyl aryl sulfonates used previously (Buelتمان, 1963). Cold-water detergents are among the newest products for the home laundry. They have a shorter hydrocarbon chain supposedly making them more effective than regular detergents at lower temperatures (Laundry Detergents,

1966).

Built synthetic detergents are the most common choice for home laundering today. These laundry detergents usually contain: 8-12% surfactants to improve the wetting ability of water, loosen, solubilize, and suspend oily soil; 20-60% complex phosphates to disperse soil particles, maintain desirable alkalinity, contribute to surfactant efficiency by reacting with fatty particles to form soap, and tie up inorganic ions to make them soluble; suds control agents, such as amides derived from vegetable oils, which act as surfactants as well as suds control agents; perfumes; soil redeposition inhibitors such as carboxymethyl cellulose (CMC); fabric brighteners which are nearly colorless dyes able to absorb ultraviolet light and reflect blue and are substantive to fabrics; and 4-8% sodium silicate for reserve alkalinity and crisp detergent granules. Other optional ingredients in detergents include carbonates (washing soda) to supplement phosphate builders, bleach, bacteriostats, blueing, and coloring to impart individuality to the product (Price, 1952, p. 71; Duthie, 1966).

Detergency

Numerous detergency studies have dealt with the variables which influence cleaning action. These variables include the detergent, the water hardness, the temperature of the wash solution, and

the type and speed of agitation (Bacon and Smith, 1954).

The detergent-soil system is complex, involving a fiber-fabric substrate of complicated physical structure to which films and particles of soil of varied and often multicomponent mixtures adhere (J.C. Harris, 1961). The ideal detergent should be able to remove all types of foreign matter deposited during soiling and prevent redeposition of these soils during laundering (Leonard, 1950).

The most recent extensive research on detergency efficiency was conducted by Galbraith (1960) who found little difference between standard synthetic detergents and standard soaps in laundering efficiency or shrinkage of wool in a home washer. She indicated that standard built synthetic detergents were superior to unbuilt synthetic detergents in cleaning action, but even the best built cold-water detergent was unable to remove as much soil from wool fabrics as standard synthetic detergents. She found that the only temperature at which cold-water detergents were superior to standard synthetic detergents in soil removal was at 70°F. More recently Consumer Bulletin (Laundry Detergents, 1966) tested four cold-water detergents and found that three gave better results in hot water than in cold water. Two of the four detergents gave satisfactory results in cold water unless the clothes were badly soiled. In a more specific study with artificial soils, Schott (1968) found that both kaolinite and calcium montmorillonite clays could be removed gradually from fabrics

with just water. Anionic detergent was able to remove about half of the remaining sodium montmorillonite from cotton. The nonionic detergent was much more effective than the anionic detergent in removing clay from this fabric.

Shrinkage of Wool Fabrics

There are three types of shrinkage which can occur during the laundering of wool fabrics. Relaxation shrinkage occurs when the fabric becomes wet and is caused by a release of strains imposed during processing of the fabric (Baird, 1961). Much of the relaxation shrinkage can be eliminated through proper finishing by steaming, sponging, etc. Felting shrinkage is caused by irreversible fiber movements resulting from agitation in water and is the most difficult to prevent. Drying shrinkage occurs when wet wool fabrics are tumble dried and can be reversed by rewetting and air drying the wool fabrics (McPhee, 1961a). Feldtman and MCPhee (1964a) state that from the consumer's point of view a fabric is unusable after it has shrunk 5-10%.

Effects of Agitation on Shrinkage

Numerous studies of the effects of agitation on wool shrinkage indicate that this is the single most important variable in wool shrinkage. Shrinkage increases with time and severity of agitation

and with increasing numbers of launderings for unfinished woolens (Furry and O'Brien, 1952; Bogaty and Harris, 1958; Feldtman and McPhee, 1964b). Any wool fabric, shrink-resistant or not, can shrink to varying degrees depending upon the washing machine, its speed and load (Feldtman and McPhee, 1964b). The most severe shrinkage occurs with impeller action washers where the water and fabric are moved by a small impeller fitted into the side of the washer (McPhee, 1961a). McPhee (1961c), Feldtman and McPhee (1964b), and Steiger (1961) have found some indication that wool fabrics shrink least in slow speed agitator washers with reversing, centrally-mounted vanes. The rate of felting of untreated wool fabrics is least in agitator washers at slow speed with a load of about four to six pounds. A load greater than six pounds produces more felting due to greater buffeting against other fabrics and the sides of the machine (Feldtman and McPhee, 1964b).

Effects of Temperature on Shrinkage

Recent studies of the effect of temperature on shrinkage of wool fabrics have indicated that the temperature is not as critical as it was formerly thought to be. Bogaty and H. E. Harris (1958) found that washing wool at 140°F. in the home washer resulted in only slightly more shrinkage than washing at 70°F. Experimentation with resin-treated wool by Feldtman and McPhee (1964a)

indicated that with increasing temperature, the rate of felting of resin-treated wool relative to untreated wool decreased slightly.

A temperature of 104-140°F. generally results in a minimum of felting for shrink-resistant wool fabrics (Feldtman and McPhee, 1964a; McPhee and Feldtman, 1961). However the dyestuff used may not be resistant to high temperature (Graham and Statham, 1959; Feldtman and McPhee, 1964a).

Effects of Detergents on Shrinkage

Galbraith et al. (1955) found that built synthetic detergents caused no more shrinkage than mild ones while removing more soil from wool. Nonionic detergents caused slightly more shrinkage than other synthetic detergents, and soaps caused slightly less shrinkage than synthetic detergents. In another study, Galbraith and Dietemann (1960) found that the type of detergent and the temperature had no significant effect on shrinkage of chlorinated wool and blended fabrics. However, standard built laundry soaps caused less shrinkage than standard built synthetic detergents, and built cold-water detergents caused almost as much shrinkage as other synthetic detergents. Increasing the wash temperature from 70°F. to 120°F. did not increase the shrinkage with soaps, but did with the cold-water detergents. Furry and O'Brien (1952) and Feldtman and McPhee (1966) have also noted that synthetic detergents are capable of inducing

higher felting rates than soaps.

Effects of Fabric Construction on Shrinkage

Generally wool fabrics with lower yarn twist and looser weave or knit structure have higher felting rates than more compact constructions (Bogaty et al., 1951; Krasny and Menkart, 1959; Farnworth, Lipson and McPhee, 1960; McPhee, 1961b; Feldtman and McPhee, 1964b). Based on a study of the effects of construction on felting shrinkage of 18 wool fabrics, Bogaty et al. (1951) further concluded that greater felting resulted from weaving the warp and filling yarns with opposite directions of twist. Twill weave wool fabrics generally felted more than plain weave fabrics. Krasny and Menkart (1959) indicated that it is possible to obtain an unfinished wool fabric with dimensional stability to mild laundering by proper attention to factors such as yarn number, twist, number of plies, warp and filling texture, and weave to impart a high ratio of compactness to the fabric. A comparison of 36 woollen and worsted weave fabrics of varying yarn count and size, weave, and weight for shrinkage when laundered ten times in a 1957 Maytag washer on mild wash for three minutes with an unbuilt anionic detergent at 100°F. indicated that there was a relationship between cover factor and percent shrinkage with higher cover area giving less shrinkage. They felt that the relationship was general enough to be used in

engineering wool fabrics to resist felting shrinkage (Bogaty, Lourigan and H. E. Harris, 1958). Hodam (1965, p. 37-49, 56-58) has reviewed other studies of variables influencing wool shrinkage.

Shrink-resistant Finishes

Over the years numerous methods of shrinkproofing wool fabrics have been used, with the most common methods attempting to reduce the differential frictional effect by partial or complete removal of the scales, or masking of the scales. These effects have been achieved by use of oxidizing agents, halogens, resins, rubbers, alkalies in dry solvents and enzymes (Sookne, 1957). An excellent review of the major methods of shrinkage control is presented in tabular form by Textile Industries (Processes, May, 1967). Further discussion will be limited to the use of polymers to prevent shrinkage.

Some processes alter the elastic properties of the wool fiber by resin crosslinking, internal polymer deposition, or spot welding (Sookne, 1957). According to Steiger (1961) an effective resin polymer for wool must possess several properties to be an effective feltproofing agent:

1. It must be a solid, but not hard and brittle,
2. it must be thermoset or link chemically to the fiber surface and,
3. it must resist removal during laundering.

Interfacial Polymerization

One of the most recent shrinkproofing treatments for wool fabrics uses interfacial polymerization (polycondensation) to protect the wool fibers, making them more abrasion- and shrink-resistant (Interfacial Polymerization, 1963). Application of a polyamide finish consists of bringing the solution of a diacid chloride in an inert, organic solvent into contact with aqueous diamine solution. The polyamide polymer is formed instantaneously at the interface of the two liquids (Whitfield, Miller and Wasley, 1961). This polyamide interfacial polymerization was developed by the Western Regional Research Laboratory of the United States Department of Agriculture, Albany, California and given the name Wurlan (Interfacial Polymerization, 1963). Wurlan, as presently used, refers to a 6-10 nylon coating on each wool fiber (Lundgren, 1967, p. 5).

Location of the Polymer. Bradbury (1963) indicated that in some cases this layer of nylon was discontinuous and produced scale masking, but in other cases it formed a complete thin layer. Electron microscopic analysis by Whitfield, Miller and Wasely (1965) revealed that the polymer was quite uniformly distributed over the surface of the fiber scales as an ultra-thin film which they estimated to be 200 to 300 Å thick. The grafting sites in wool are free amino groups of N-terminal amino acids and amino and hydroxyl groups

of side chains in the wool protein. There also appeared to be a preferential deposition of the resin on the wool fiber tips indicating that the nature of the deposition may be influenced by the fabric construction (Fong et al., 1962).

This polymer finish increases fiber friction in both directions of the fiber and considerably masks the scales of the fiber, restricting fiber migration. However, it is not known if this is the only mechanism involved in the shrinkproofing process (Whitfield et al., 1965).

Performance of Wurlan Finished Wool Fabrics. A polymer shrink-resistant treatment gives better resistance to felting under severe conditions than other types of shrinkproofing processes (Feldtman and McPhee, 1966). Plant trials of Wurlan treated fabrics by Fong, Ash and Miller (1963) gave quite a variation in felting shrinkage depending upon the laundry conditions. A multicycle mild wash followed by tumble drying resulted in from 0.8-4.0% shrinkage after five launderings. Variations in shrinkage depended upon the exact finishing application and the fabric weave, with twill shirting shrinking more than plain weave shirting of similar weight. They concluded that this was adequate resistance to felting shrinkage since the mill control limit is 3% for washable woolens. In a more recent plant trial, Miller and Fong (1965) indicated that modifications in equipment and processing allowed for more uniform shrinkage

resistance with less than 4% in the warp direction and none in the filling direction after four 75-minute top loading agitator washes.

Two studies have indicated that the effectiveness of the Wurlan finish is closely allied to the fabric structure. Hine, Lipson and McPhee (1964) noted that polymerization produces better shrinkage resistance for woolens than for worsteds possibly because the woolens have a higher percentage of fiber tips which can be coated. Fletcher and Roberts (1965) found that wool knits treated with Wurlan finish did not achieve satisfactory shrink resistance. Recently seven shrinkproofing treatments were compared in an international round-robin test supervised by The German Textile Research Laboratory. This comparison indicated that Wurlan finished wool gave the best shrinkproofing under rigorous washing conditions (Lundgren, 1967, p. 6).

Advantages of the Wurlan finish which have been noted include ease of application to a wide range of fabrics, increased wet wrinkle recovery and no alteration in dry wrinkle recovery, slight stiffness along with higher tensile strength, increased air permeability and greater resistance to pilling (Fong et al., 1962). Whitfield, Miller and Wasley (1961) found slightly different results with no significant difference between Wurlan finished and unfinished wool in handle (flexural rigidity), wrinkle recovery, color, breaking strength, and percent elongation. A slight increase in resistance to acids,

alkalies, and oxidizing agents was also noted. Fong et al. (1962) reported that the polyamide finish also improves the appearance of the wool fabric after laundering.

Feldtman and McPhee (1964c) treated plain weave woolen fabric with a polyamide resin and found that a two percent resin finish increased abrasion resistance by 50%. However, plant trials by Fong, Ash and Miller (1963) indicated a lower resistance to flexural abrasion (Stoll) and a lower tear resistance (Elmendorf tear) for Wurlan finished fabrics.

Market Success of Wurlan Finished Wool Fabrics. Presently in the United States, Wurlan finished wool fabrics are being sold by J. P. Stevens as " H_2O " fabrics and by Joseph Bancroft and Sons as "Bancora" (Washable Wools, 1964). Four major chemical companies are now marketing polymer-type shrink-resistant treatments, and leading textile equipment manufacturers are developing appropriate machinery (Machine Washable Wool, 1967).

The acceptance of washable woolens by consumers has been disappointing. This may be due to past experience with washable woolens. Also a full range of colors for the home sewer is not presently available, and very few ready-to-wear articles are being offered to the consumer (Smith, 1966).

Recent Modifications of Wurlan Finish. The most recent approach to interfacial polymerization of wool is the application of

preformed reactive polymers by cross-linking with another reagent. This method is known as phase-boundary-limited crosslinking (PBLC) or Wurlan II. This finish has the advantages of being cheaper, just as effective, and extending the range of wool-containing products which can be successfully finished by interfacial polymerization. Recently a cheaper diamine, ethylene diamine, has been substituted in the polyamide finishing reaction. This diamine improved the dimensional stability and hand of knits and was expected to be on the market by fall of 1967 (Lundgren, 1967, p. 7-8).

Breaking Strength of Wool Fabrics

Breaking strength tests on unfinished lightweight wool challis by Furry and O'Brien (1952) indicated a slight loss of strength and slightly less uniformity in strength after repeated laundering, with greater agitation causing greater loss of strength as well as greater felting shrinkage. However, after laundering wool blankets without agitation for ten launderings, Petzel et al. (1961) found a significant increase in the warp grab breaking strength possibly because of the felting shrinkage.

Whitfield, Miller and Wasley (1961) found that the cut strip breaking strength of unlaundered Wurlan finished wool flannel fabrics was similar to unfinished wool flannel fabric, but later work by Fong et al. (1962, 1963) indicated that the tensile strength of plain weave flannel wool fabrics was slightly increased by the Wurlan finish. No breaking strength tests after laundering have been

located in the literature.

Recommendations for Laundering of Wool Fabrics

Recommendations for laundering of wool vary and are somewhat dependent upon the type of equipment and laundry products available at the time. There is still some disagreement over the detergent which should be used to launder wool. The laundering instructions for "H₂O" machine washable woolens sold by J. P. Stevens and Co., Inc. are: machine wash with low agitation, warm temperature, use mild soap and place on a hanger to drip dry. Feldtman and McPhee (1964a, 1964b, 1966) stated that, based on their research, laundering conditions to minimize felting include slow speed agitation, high pH and temperature, and soap rather than an anionic or nonionic detergent. Olson (1965) also recommended gentle agitation and warm water but suggested that standard built detergent was necessary for better soil removal. Forecast for Home Economics (The Laundry Load, 1967) recommended that machine washable wool be laundered with standard, built synthetic detergent or soap with a fabric conditioner or water softener, at warm wash temperature with gentle or no agitation for 3-6 minutes. Then the articles should be spun at fast speed for a short period of time, followed by regular drying for 10-15 minutes until partially dry with dry towels as buffers in the dryer. Based on extensive research,

Galbraith (1955, 1960) recommended a temperature of 120°F. for optimum cleaning efficiency of wool for all detergents including the cold-water types for as short a time as possible. Taube (1965, p. 10, 17) recommended cold water (80°F. or less) for lightly soiled wool items and fabrics that lose color, wrinkle excessively, or shrink in hot water.

It is generally recommended that wool fabrics be taken out of the dryer after all loose water is removed, but before the water regain is reduced significantly below normal saturation regain (Feldtman and McPhee, 1964b). The dryer instruction booklet noted that most woolens should not be tumble dried unless the manufacturer of the fabric suggests it (Whirlpool, 1967). However, experimentation with wash-and-wear woolens by Krasny and Menkart (1959) indicated that if fabrics were dimensionally stable to laundering, tumble drying did not produce more shrinkage. They also indicated that fabrics were less mussed when tumble dried than when air dried. Further recommendations and studies have been reviewed by Hodam (1965, p. 53-55).

The information gained from this review of literature pertaining to soils, their transfer to fabrics, removal from and redeposition onto fabrics was useful in determining the type and amount of artificial soil to apply, the application of the soil and laundering conditions optimum for removal of that soil. A survey of the

common methods of detection of soil on fabrics revealed the advantages and disadvantages of each method.

Literature on shrinkage and laundering of wool fabrics was valuable in preparation of the specimens prior to laundering and determination of the optimum conditions for laundering and drying. A description of the Wurlan finishing process and its location on the fiber gave some indication of the durability and soiling tendencies that might be expected. The review of previous research on Wurlan finished wool fabrics was a necessary aid in the choice of a problem which would contribute new information about the performance of these fabrics.

PROCEDURE

Soil Retention of Wool Fabrics Laundered with Gentle or No Agitation

The amount of dibromostearic acid and kaolinitic and micaceous clay retained by artificially soiled wool fabrics after laundering was determined by X-ray fluorescent spectroscopy of bromine in the oily soil and aluminum in the clay. The laundering conditions were gentle or no agitation and a standard, anionic detergent at 120°F. for five minutes.

Materials

Fabrics. Wool fabrics were secured from the J. P. Stevens Co., Inc., Dublin Plant, Dublin, Georgia. The unfinished plain and twill weaves were greige goods, while the Wurlan finished plain weave was white and the Wurlan finished twill weave fabric was navy blue. Appendix A contains swatches of these fabrics.

Soil. The soil chosen for this study consisted of Spinks Bandy Black Research Clay obtained from the Spinks Clay Company of Paris, Tennessee and a mixed oil containing dibromostearic acid, oleic acid, cholesterol, cetyl alcohol, squalene, tristearin and triolein.

The Bandy Black Clay was used because it is predominantly

kaolinite which is one of the most abundant clay minerals in nature. Tuzson and Short (1962) found an excellent reflectance correlation between the removal of this clay and the removal of natural soil from fabrics.

The mineralogical content of the Bandy Black Clay, as determined by the H. C. Spinks Clay Company, was approximately 80% kaolinite, 14% silica content, and about 3-4% illite (mica).¹ Mineralogical analysis by G. A. Borchardt indicated that this total "clay" was approximately 50% clay and 50% silt. X-ray diffraction indicated that the clay fraction (less than two microns in diameter) consisted of a large percentage of kaolinite and smaller amounts of mica as well as very small amounts of rutile and anatase identified according to a procedure developed by Raman and Jackson (1965). The chemical formula for the clay minerals present is given in Chapter I.

Kennedy and Stout (1968) listed the chemical analysis of this clay as:

Silicon Dioxide	61.00%
Aluminum Oxide	24.54
Iron Oxide	0.99
Manganese Oxide	0.01
Titanium Dioxide	1.29

¹Paul Turbett, Director of Engineering and Research, H. C. Spinks Clay Company. August 29, 1967.

Calcium Oxide	0.02
Magnesium Oxide	0.12
Sodium Oxide	0.36
Potassium Oxide	1.69
Sulfur Trioxide	0.11
Phosphorous Pentoxide	0.07
Loss on ignition	9.74

Mineral and chemical analyses were necessary to determine which element could be used as representative of clay retained by these fabrics. Elements which were considered for possible analysis included iron, silicon, titanium, and aluminum. As noted in Chapter I, silicon dioxide is found in the kaolinite and mica portions of the clay while iron oxide is located in the mica. Rutile and anatase consist of titanium dioxide. Kaolinite and mica both contain aluminum which is one of the lowest atomic weight elements presently detected by X-ray fluorescent spectroscopy.

The Bandy Black Clay was separated into silt (large particles) and clay (particles less than two microns in diameter) with the clay being used for this study. It was desirable to limit particle size for this study because the percentage of most elements in the soil may change with the particle diameter.

To separate the silt from the clay, 100 gms. of soil were suspended in 1,000 ml. of distilled water with sodium carbonate (5.3 gms/

100 ml. H_2O) to give a pH of 9.5-10.2. The suspension was stirred vigorously and allowed to settle 7.1 hours for every 10 cm. of depth at 24°C. or 7.0 hours for 10 cm. of depth at 25°C. (Jackson, 1956, p. 114-115). The clay suspension was then decanted carefully so that the silt that had settled out was not disturbed. Adding excess sodium chloride to the clay suspension resulted in complete flocculation of the clay in approximately 12 hours. The clear liquid on top of the clay was poured off, and two techniques were then used to dry the clay. In the first procedure, the clay was centrifuged to dry it as much as possible and was then freeze dried, causing the clay to dry in platelets. The second technique consisted of drying the clay in metal pans on the hot plate (170°C.), after the flocculation, followed by grinding in a Waring Blender. It was hoped that freeze drying would eliminate grinding to break up lumps. However, the platelets formed by freeze drying were more resistant to grinding with the use of both a ball mill and a mortar and pestle than clay dried with heat. Clay dried by both procedures was sifted through a 0.250 mm. mesh sieve and mixed together thoroughly to assure an even distribution of particle sizes. The moisture content of the clay was allowed to come to equilibrium with the room humidity. Approximately 1,000 gms. of clay were prepared for the entire study.

The dibromostearic acid was obtained by adding bromine across

the double bond of oleic acid to provide an element in the oil which could be detected with X-ray fluorescent spectroscopy. Theoretically, it is possible to determine when all of the bonds of the oleic acid have been saturated with bromine by the persistence of a brown color. However, preliminary work indicated that the visual end point was not distinct enough to actually do this. It was calculated that 45.25 gms. of bromine per 79.75 gms. of oleic acid would give the desired product containing two atoms of bromine for each molecule of oleic acid. The dibromostearic acid was prepared by weighing out the oleic acid, followed by addition of the bromine dropwise with continual stirring until brominated oil weighed 125 gms.

A mixed oil was used because it is similar to the composition of sebum (Brown, 1947; Powe and Marple, 1960). It was prepared by mixing the liquid oils [triolein (14.7% by weight), oleic acid (8.8%), squalene (4.9%), and dibromostearic acid (50.0%)] in a large beaker. The solid components [cetyl alcohol (4.1%), cholesterol (2.9%), and tristearin (14.7%)] were gently heated in a beaker until melted. These oils were then added to the liquid mixture according to a procedure developed by Richards (1966, p. 33). This was not the most satisfactory procedure since the melted oils solidified as they poured into the cooler liquid mixture. Five liters of perchlorethylene were added to the 250 gms. of mixed oil to keep it liquid. This amount of oily soil was sufficient for the entire study.

Methods

Fabric Description. Alfred Suter Co. Textile Instruments were used to determine the yarn count and yarn twist according to ASTM Standards D1910-64 and D1422-65T (1966).

Fabric Preparation. No test swatches were cut within 10% of the width of the selvage edge (ASTM standards D1910-64). Three 5 × 5 in. test specimens were cut from each type of fabric for each of three laundering intervals (10, 15, and 20) for the two laundering conditions of gentle or no agitation with a standard anionic detergent at 120°F. Replacement specimens for intervals 10 and 15 were also cut. For each laundry condition, there were nine test specimens plus six replacement specimens for each of four types of fabric. A soiling check for each type of fabric was cut for the 1st, 10th, and 20th soil applications to give a total of 12 soiling check specimens. Another 12 specimens for each load were cut to be laundered but not soiled, and four of these specimens (one of each type of fabric) were withdrawn along with the soiled and laundered specimens after launderings 5, 10 and 20. Holders for laundering these 5 × 5 in. specimens consisted of strips of Wurlan finished plain weave fabric measuring 6 × 35 in. Twelve specimens were attached to the edges of each holder with two pins per specimen. To bring the weight of the wash load to $4\frac{1}{2}$ pounds, buffer fabrics measuring approximately

22 × 22 in. were cut. A total of 11 buffer pieces consisting of two unfinished plain weave and three each of the other three types of fabrics were used. The test specimens, holders, and buffer fabrics were finished with a wide zig-zag stitch using colored mercerized thread as a code. Threads were attached along the middle of each edge of the soil test specimens to serve as handles for dipping the fabrics into the oil-perchloroethylene solution. All fabrics were number coded with a laundry marking pen.

Application of Soil. The mixed oil was prepared by pipetting 15 ml. of the oil and perchloroethylene solution previously described into an eight-inch square pyrex cake pan. One hundred thirty-five ml. of perchloroethylene were added to the solution and mixed thoroughly. Specimens were dipped horizontally into the solution for ten seconds, allowed to drip vertically from the center of opposite sides of the specimen for ten seconds, turned 90 degrees and allowed to drip for another ten seconds.

The soiling solution described above was sufficient to soil six specimens, and resulted in application of approximately 2% oil. The specimens were then dried on a "Fiberglas" screen under the hood for two hours and allowed to age for at least 20 hours before clay was applied.

Dry clay was applied to the fabrics by tumbling in one-quart steel cans in an Atlas Launder-Ometer for ten minutes. Each can

contained ten steel balls, the clay, and one fabric specimen. An excess of clay was measured into the cans using a plastic container holding about 0.42-0.45 gms. of unpacked clay. The clay was stirred thoroughly prior to measurement, scooped out, gently dropped into the plastic holder, and then leveled off at the top. Weight trials of the accuracy of measurement by this method indicated a variation of approximately 3.5% of the weight of the clay. This was sufficiently accurate since specimen weights were more variable. After application of the clay, the specimens were shaken to remove excess clay and vacuumed with low suction using the upholstery attachment with a Hoover Portable Vacuum Cleaner. Visual examination indicated that soil was applied uniformly by this technique. This was verified by preliminary X-ray fluorescent spectroscopy. The average application of clay was 1.9% by weight of the fabric.

There were 36 specimens per laundry load to be soiled, except for the soil check intervals when there were 40 specimens. This required that two Launder-Ometer loads be run. After the first load, the cans and covers were wiped out with a clean, dry cotton cloth. After the second load each day, the covers were wiped out with a damp cloth and the cans were washed and rinsed. Once a week the covers were separated from the rubbers and washed.

Laundering Conditions. The specimens attached to holders, along with 12 shrinkage specimens (22 × 22 in.), and buffer fabrics

to give a weight of $4\frac{1}{2}$ lbs. were laundered with $1\frac{1}{4}$ cups of detergent at 120°F. for five minutes with either gentle or no agitation. A rinse temperature of 104°F. and a high spin were used for both laundry conditions. A standard built anionic detergent, "Tide," which was purchased on the open market (Jan., 1968) in a single lot, was used to launder these wool specimens.

The specimens were laundered in a Whirlpool Imperial Mark XII, a 1968 model multicycle washer with a reverse action centrally mounted agitator. The dryer used was also a 1968 Imperial Mark XII equipped with a drying rack that remained stationary as the dryer drum turned. This rack was recommended for drying wool socks and sweaters, etc. The soiled and laundered specimens were dried on the rack at high temperature for 15 minutes (five minutes at 127-147°F., ten minutes of cooling) with the specimens pinned to the holder, doubled over so that no fabric was hanging over the edges of the rack. The specimens were slightly damp after this drying procedure, and were allowed to air dry before being soiled again.

Soil Detection. The specimens were prepared for soil analysis by cutting a $1\frac{1}{4}$ inch diameter circle from the center of each five-in. square specimen with an Osborne arch punch. These were then applied to circular plastic disks of the same size with Elmer's glue to allow for use of vacuum X-ray fluorescent spectroscopy without

a mylar retaining window to keep the specimens in the holder. This was desirable because the count rate resulting from excitation of low energy elements such as aluminum and silicon is greatly reduced by a mylar window.

A Philips X-ray Fluorescence Spectrometer with a tungsten target X-ray tube operated at 50 kilovolts, 35 milliamps and a lithium fluoride analyzing crystal were used with a scintillation counter to detect the bromine of the dibromostearic acid on the wool specimens. Aluminum of the kaolinite and mica in the clay was detected using a chromium target X-ray tube operated at 50 kv. and 40 ma. with an ethylenediamine ditartarate analyzing crystal. The detector used for aluminum was a flow counter with 90/10, argon/methane counting gas. The specimen chamber was evacuated to reduce absorption of X-rays by air and thus increase the count rate.

Several techniques were used to allow for comparison of specimens read on different occasions. It was not possible to set the X-ray tube on exactly the same current every time so one bromine specimen was counted to get an average of three observations every time the X-ray tube was turned on. Voltage and amperage were adjusted accordingly to give a count rate as close as possible to the count rate observed on a previous occasion for that specimen. This involved collection of 381×10^3 counts on each occasion. Counting of aluminum under vacuum varied slightly for each group of four

specimens, requiring that one specimen be used as a standard with every load to correct for these vacuum differences.

For all specimens, a fixed count rate which would give at least one-minute counts was selected. For bromine, 32×10^3 to 128×10^3 counts per observation were collected depending upon the amount of oil present, with two determinations for each specimen. Since the count rate was high for this element and the background count, taken at an angle one degree below the peak, was less than 10% of the peak count, no background count was taken (Jenkins and DeVries, 1967, p. 96). The count rate of aluminum was much lower so that 8×10^2 to 32×10^2 counts per observation with two or three observations were taken to give at least one-minute observations. Background counts, blank specimens, and redeposition specimens had such low count rates that 4×10^2 counts were collected per observation with two or three observations to give a total of about one minute of counting time. For aluminum, the background counts were quite substantial in relation to the aluminum peak counts. Therefore, the backgrounds were counted at an angle of one degree below the peak angle of 2θ .

All aluminum determinations were based on peak to background ratios which had been adjusted for vacuum variations and for differences in shrinkage based on weight ratios of the unlaundered and laundered specimens. Bromine content was based on counts per

second with adjustment again made for weight ratio shrinkage. It was necessary to use a shrinkage factor because of shrinkage of up to 25% for some specimens which resulted in an increase in the area of fabric exposed to X-rays. The percentage of bromine and aluminum on laundered fabrics was computed by comparison to count rates of fabric standards with known amounts of oil and clay.

Preparation of Clay and Oil Standards. Untreated specimens were assumed to have zero concentrations of oil and clay. Blank specimens with count rates below the average were usually within the experimental counting error (0.56% for oil and 1.09% for clay) particularly for oil. Some deviation of the aluminum counts on the blanks also may be explained by the presence of contaminants.

Wool fabric standards of varying oil concentrations were prepared by pipetting a known quantity of the mixed oil onto $1\frac{1}{4}$ in. diameter specimens to give five replicates of 1.0, 2.0 and 4.0% by weight of the fabric. This technique produced standards with little error.

Preparation of clay standards was more difficult, principally because of the low concentrations required and the tendency of the clay particles to migrate to the edges of the specimen which could not be exposed to the X-ray beam. Initially small amounts of clay were weighed out, suspended in water, and pipetted onto $1\frac{1}{4}$ in. diameter fabric specimens. As was noted above, clay migrated to

the edges of the fabric, and also was deposited on the glass instead of the fabric. The small amounts weighed out were not accurate enough and it was difficult to keep the clay particles in suspension as it was being pipetted.

The second procedure used to develop clay standards consisted of the use of a circle of fabric five times the area of the specimen needed, to which five times as much clay as desired for the standard was applied. This reduced the edge exposure in relation to the area to minimize the effect of migration to the edges. The specimens were first wired to horizontal rings with plastic coated wire in six spots around the circle so that the specimens were not in contact with anything upon which the clay could be deposited. The clay suspensions were prepared to give the concentrations desired, but at a weight that could be determined more accurately (100 times the aliquot needed). The standard clay suspensions consisted of the weighed amount of clay, 250 ml. of water, 50 ml. of ethanol to aid in suspension, and a small amount of sodium carbonate to raise the pH for temporary clay suspension. These components were placed in a ground glass stoppered flask in the order given and the contents were thoroughly shaken prior to pipetting the clay suspension evenly onto the surface of the fabric. Five replicates of concentrations 0.125, 0.25, 0.50, 1.0 and 2.0% by weight of the fabric were prepared. A large number of clay standards were needed

because the replication error was rather large. After the clay had dried, $1\frac{1}{4}$ -in. diameter circles were cut from the center of each of these larger circles.

Dimensional Change and Breaking Strength of Wool
Fabrics Laundered with Standard and Cold-Water
Synthetic Detergents at Gentle or No Agitation

The dimensional change and breaking strength of unfinished and Wurlan finished plain and twill weave wool fabrics after 20 launderings have been compared using two agitation speeds and two detergents at 120°F. for five minutes.

Materials

Fabrics. The fabrics described in Table 1 were compared for dimensional and breaking strength changes caused by laundering.

Detergents. The standard anionic detergent, "Tide," described previously was compared to the liquid nonionic detergent, Cold Water "all" which was secured in a single lot from Lever Brothers.

Methods

Fabric Preparation. Three shrinkage specimens for each type of fabric were cut 22 × 22 in. randomly from the samples (ASTM Standards D1284-59) for each of four laundering conditions:

1. no agitation with standard detergent,
2. gentle agitation with standard detergent,
3. no agitation with cold-water detergent, and
4. gentle agitation with cold-water detergent.

Five specimens for the warp grab breaking strength were cut 4×8 in. according to ASTM Standards D1682-64. The breaking strength specimens for the laundered fabrics were taken from the center of the shrinkage specimens after the dimensions were determined at the end of 20 launderings. The laundry loads for the shrinkage tests with the cold-water detergent required four extra pieces of buffer fabric (two unfinished twill weave fabrics and one each of the Wurlan finished fabrics). The shrinkage and buffer fabrics were finished with a long, wide zig-zag stitch and coded with different colors of mercerized cotton thread. All fabrics were also number coded with a laundry marking pen.

Relaxation of Shrinkage Specimens. The shrinkage specimens were relaxed prior to laundering by immersion in a solution of 0.1% Nacconal for at least five hours in $100^{\circ}\text{F}.$ water which cooled to room temperature during the relaxation period (ASTM Standards D1284-59). Excess water was extracted by spinning in the washer for several minutes followed by drying the specimens on a flat counter. After being conditioned for at least eight hours, the specimens were marked in three spots in both the warp and filling directions with

a laundry pen using a cardboard template which measured 45 cm. square. The navy blue, Wurlan finished twill weave fabric was marked with white thread.

Laundry Conditions. There was a total of four loads, each containing three shrinkage specimens for each of four types of fabric samples. Shrinkage specimens were laundered along with the soil specimens and buffer fabrics under the two conditions given earlier for the soil specimens. One-half cup of Cold Water "all" was added to the two loads laundered with this detergent. The shrinkage specimens were removed from the washer immediately after the laundry cycle and laid out to dry without stretching on a flat, nonabsorbent surface.

Measurement of Dimensional Change. The dimensions of the shrinkage specimens were determined after 1, 5, 10, and 20 laundrings. Three measurements in each direction were taken after conditioning at standard conditions ($70 \pm 2^\circ\text{F.}$, $65 \pm 2\%$ relative humidity) for at least eight hours.

Measurement of Breaking Strength. Warp grab breaking strength tests were performed according to ASTM Standards D1682-64. All specimens were held at standard conditions for at least eight hours prior to testing. A Scott Pendulum Tester Model J (constant rate of traverse) with jaws of the front clamps measuring one in. wide by one in. high and jaws of the back clamp measuring

three in. wide by two in. high was used. The distance between the clamps at the start of the test was three in. and the speed of the traverse was 5-11 seconds to produce an acceptable break. The warp breaking strength was determined as an average of five successful breaks. No breaking elongation was determined with the grab test because it is not appropriate since there is a lack of uniformity in elongation between the yarns fastened within the jaws and the adjacent yarns (Kaswell, 1963, p. 471).

RESULTS AND DISCUSSION

Fabric Description

The unfinished and Wurlan finished twill weave fabrics used in this study were similar in yarn count and yarn twist (Table 1). However, the Wurlan finished plain weave was lower in both yarn count and yarn twist than the unfinished plain weave even though both fabrics had the same lot number. The yarns in all fabrics were single yarns twisted in the S direction.

Table 1. Yarn count and yarn twist of wool fabrics.

Fabric	Yarns/inch*		Twist/inch**	
	Warp	Filling	Warp	Filling
Unfinished griège, plain weave	36	37	29	29
Wurlan finished, plain weave	33	29	22	24
Unfinished greige, twill weave	35	30	24	23
Wurlan finished, twill weave	35	32	23	22

*ASTM D1910-59T, 72°F. 65% relative humidity

**ASTM D1422-59T

Soil Retention of Wool Fabrics Laundered with Gentle
or No Agitation with a Standard, Anionic Detergent

Results of Preliminary Experimental Work

Problems related to the soil composition, soiling technique, and amounts of soil picked up by Wurlan finished and unfinished wool fabrics were investigated prior to the final study.

Soil Composition. A comparison of the removal of dibromostearic acid alone to that of a mixed oil containing 20% dibromostearic acid tentatively indicated that removal of the dibromostearic acid from wool fabrics was not influenced by the presence of the other oils. However, the variation between replicates soiled and laundered only once was large enough to obscure any possible differences. A mixed oil described in the previous chapter was used for the study since it was unknown what effect, if any, the composition of the oil might have upon the removal of clay or the interaction of different oils with the Wurlan finish.

Soiling Technique. It was desirable to develop a method of applying oil to these fabrics which would give maximum uniformity. Specimens were soiled by horizontal immersion in the oil, perchlorethylene solution for ten seconds followed by four methods of draining the excess. Four methods to remove the excess oil were tested: a vertical drip for 15 seconds supported by pick needles at one end of the specimen (Oregon. State University, 1968); a vertical drip from the center of the specimen with a 90 degree reverse after ten seconds to drip for another ten seconds; absorption of excess oil

and perchlorethylene by a paper towel for ten seconds; and a vertical drip supported by pick needles at one end of the specimen for ten seconds followed by a 180 degree reverse and drip for another ten seconds (Richards, 1966, p. 34). The paper towel technique resulted in poor uniformity and the vertical drip with 180 degree reverse of the specimen was even worse. The most uniform application was achieved by dripping from the center of the specimen with a 90 degree reverse, but the use of pick needles at one end of the specimen with no reversal was almost as uniform. Vertical dripping from the center of the specimens with a 90 degree reverse was used for this study.

X-ray fluorescent spectroscopy of preliminary applications of the mixed oily soil indicated that replicates picked up equal amounts ($\alpha = .05$) and that weave and finish did not affect the amount of oil picked up by these wool fabrics ($\alpha = .05$).

Since the procedure for dry soiling of these wool fabrics with clay was not described in the literature, it was necessary to determine the degree of variation of soil picked up by replicates and the uniformity of application to any one specimen. These data were necessary to determine the specimen size, whether or not one circle from each specimen would be representative of that specimen, and the number of replicates needed.

Uniformity of application of clay to single specimens was

determined by X-ray fluorescent spectroscopy of iron. There was little variation among three circles from single specimens.

Analyses of two specimens of each type of fabric for clay application (iron) indicated no difference between duplicates ($\alpha = .05$), or among the different fabrics. While the differences in application were not significant, clay applied to these fabric specimens varied by 16.6%. This may seem like quite a large variability to accept. However, it should be noted that only about 1/60 to 1/400 of the clay applied was retained after 20 launderings. Also, the variation in clay retained would be greatly reduced by 20 applications. Both Sanders and Lambert (1950) and Berch et al. (1967) found a similar large variation for application of dry clay to fabrics. Aqueous application of a montmorillonite clay by Richards (1966) resulted in a lower variability of 5.6%.

Based on these preliminary results of application of clay, specimens measuring five inches square were used with only one circle from each specimen being analyzed for soil. Three specimens were used for each determination because of the rather large variation in application of clay to replicates.

Comparison of the Elements of Spinks Bandy Black Clay by X-ray Fluorescent Spectroscopy. Preliminary X-ray fluorescent spectroscopy of the various elements (Appendix B) of this clay indicated that iron would give a higher count rate than other elements.

However, this element is not present in kaolinite which is the chief constituent of the clay (> 80%). Also, iron oxide is a rather common contaminant. The unlaundered fabric specimens varied more in content of iron than other elements even between specimens from different areas of the same fabric. For these reasons, iron was not the most desirable element to use as an indicator of residual clay content.

Titanium also resulted in high X-ray fluorescent spectroscopy count rates. However, this element is found in the rutile and anatase minerals which are just a small fraction of the total clay.

Micas may contain aluminum and magnesium, as well as iron. The mica in this clay probably contains all three of these elements. Silicon and aluminum are found in both kaolinite and mica, accounting for more than 90% of the clay. These two elements should be very good indicators of clay retained by fabrics. However, both elements produce soft X-radiation (lower limits of detection) resulting in low count rates.

The element to be used as a tag for the clay was not chosen until the completion of the 20 launderings, at which time a ratio of the count rates of three elements on ten specimens (unlaundered, laundered, and soiled and unlaundered) was determined. There was a constant ratio of silicon to aluminum content with a large fluctuation in titanium content. This seemed to indicate that rutile and

anatase might be removed in a different manner, and that titanium would not be a suitable indicator of the removal of total clay. This finding indicates that it is desirable to determine what clay minerals are present, and in what proportions prior to use of a single element as a tag for clay. Both silicon and aluminum could be used as indicators of the removal of this Bandy Black Clay. Aluminum was chosen because it resulted in slightly higher count rates.

Results of the Final Study

X-ray Fluorescent Spectroscopy--Detection of Clay and Oil

Standards. A discussion of the preparation of standards has been given in the previous chapter. The clay and oil retention of laundered specimens was determined by plotting the count rate of the standards containing a given percentage of soil by weight of fabric. The result was quite successful for oil; correlation coefficient, $r = .995$ for the standard curve (Figure 1). Peak to background ratio versus the percentage of clay by weight of fabric resulted in a standard curve with the averages of five replicates producing a relatively straight line (Figure 2). In this case it was observed that the replication error increased with increasing concentrations so that there would be greater confidence in values occurring at the lower end of the curve. The regression coefficient for this line ($r = .91$) was satisfactory, although there would be some room for improvement of

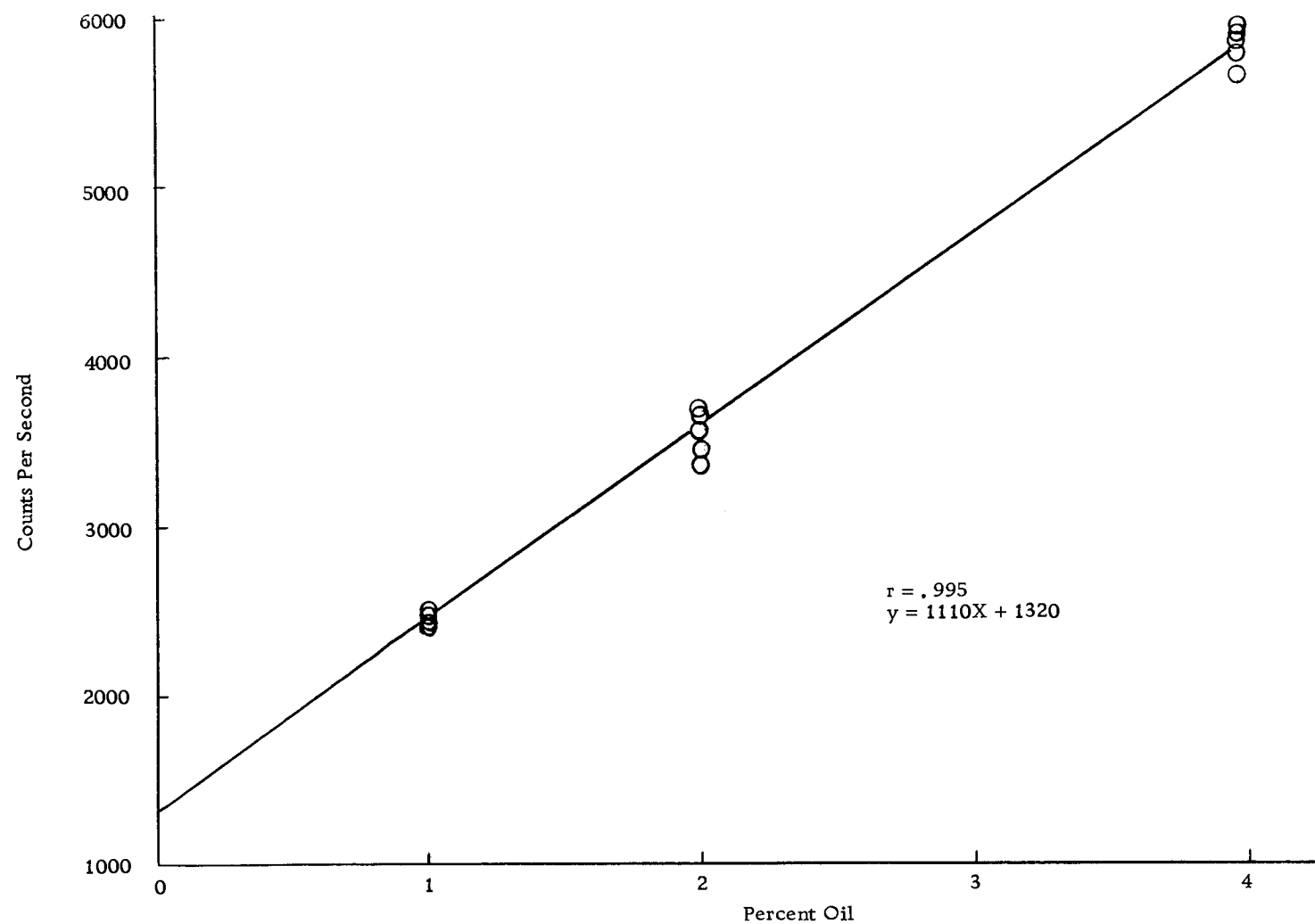


Figure 1. Standard curve: percentage of oil.

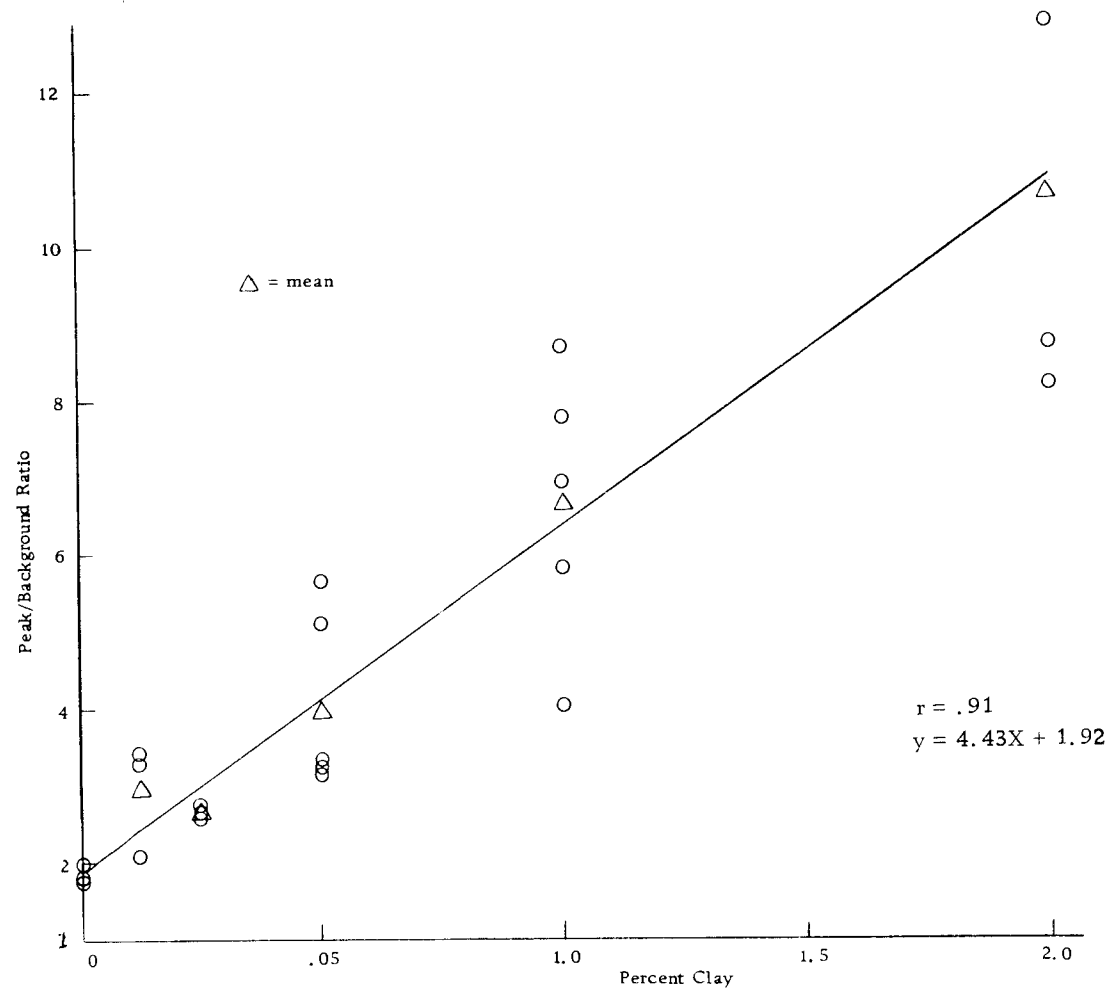


Figure 2. Standard curve: percentage of clay.

variability between replicates. Data for statistical analyses of soil retention and Figures 3, 4 and 5 were based on these standard curves.

Application of Soil. X-ray fluorescent spectroscopy of aluminum indicated that the average application of clay was 1.92%. Both plain weaves retained 1.85%, the unfinished twill picked up 1.89%, and the Wurlan finished twill picked up 1.96% clay. The average amount of oil picked up by these fabrics was 2.06%. Unfinished plain weave fabrics retained 1.60%, Wurlan finished plain weave 1.70%, unfinished twill weave 2.21%, and Wurlan finished twill weave 2.80% oil. These averages are based on three specimens soiled at different times.

The variation in application of soil over the period of this study was determined by X-ray fluorescent spectroscopy of a specimen of each type of fabric soiled at applications 1, 10, and 20. The averages of these four specimens from each interval were compared for significant variation by a t test. There was no difference in application of either oil or clay ($\alpha = .05$) at the various intervals. As was noted earlier, the variation in amount of clay applied to specimens was quite high (15.4% over the period of the study) with no differences among the fabrics.

The method used to apply oil has been used in several other studies (Richards and Morris, 1968; Oregon. State University, 1968).

While this method is not very similar to the natural application of body oils, a more realistic and easy way to apply artificial oil uniformly to fabrics has not been developed. Dry soiling with clay was quite satisfactory for these wool fabrics. It was felt that this method of application is representative of the kind of particulate soiling which most commonly occurs. With the difference in chemical nature of the unfinished and Wurlan finished wool, this method resulted in similar amounts of clay application, while a liquid immersion method might be expected to give some difference due to a small difference in water absorption. Soiling checks of bentonite montmorillonite applied by liquid immersion to a 65/35 mohair/wool blend with and without Wurlan finish did indicate that slightly more clay was picked up by the unfinished fabric (Richards, 1966, p. 60). However, this tendency would need to be verified by a larger sample. While the dry application of clay used in this study was satisfactory, there is some merit in further work on a method similar to the one proposed by Berch et al. (1967) to reduce the time involved in application of the soil.

Detection of Differences in Soil Retention by Wool Fabrics

After Laundering. The average percentage of clay and oil retained by these wool fabrics is given in Table 2. The lower limits of detection of clay retained by fabrics in this study was 0.09% using X-ray fluorescent spectroscopy. It would be possible to detect smaller

Table 2. Average percentage of clay and oil retention indicated by X-ray fluorescent spectroscopy.

Interval	-----Fabric-----							
	Unfinished plain weave		Wurlan, plain weave		Unfinished twill weave		Wurlan, twill weave	
	Clay	Oil	Clay	Oil	Clay	Oil	Clay	Oil
<u>Gentle agitation</u>								
10 (avg.)	-0.04	0.72	0.05	0.70	-0.08	0.34	0.11	0.76
15 (avg.)	-0.04	0.75	0.04	0.85	-0.13	0.34	.21	0.74
20-a	-0.10	0.77	0.08	0.90	-0.11	0.51	0.09	0.80
20-b	-0.055	0.85	0.105	0.84	-0.08	0.55	0.125	0.99
20-c	-0.11	0.87	0.12	1.28	-0.07	0.45	0.14	0.78
20 (avg.)	-0.09	0.82	0.10	1.00	-0.09	0.50	0.12	0.88
<u>No agitation</u>								
10 (avg.)	0.26	2.02	0.58	2.42	0.07	1.85	0.37	3.56
15 (avg.)	0.27	2.42	0.49	2.13	0.01	1.92	0.49	3.00
20-a	0.26	2.88	0.515	2.25	0.035	2.03	0.91	2.23
20-b	0.27	2.73	0.56	3.14	-0.015	2.00	0.49	3.21
20-c	0.26	2.80	0.66	2.88	0.02	2.69	0.51	4.19
20 (avg.)	0.26	2.81	0.58	2.76	0.01	2.24	0.64	3.88

amounts of clay by counting longer or detecting an element with higher count rates to reduce the standard deviation of counting. Higher count rates for bromine resulted in a detection limit of 0.025% for oil. Hypothesis 1 has been accepted since this method can detect small amounts of soil retained by fabrics.

Table 2 and Figures 3 and 4 reveal that a larger percentage of oil than clay was retained by all of these wool fabrics. Based on three standard deviations of counts collected for these determinations, there was no difference between the clay content of Wurlan finished plain and twill weave fabrics after 20 launderings under both conditions, although more clay was retained when no agitation was used. There was also no difference in oil retained by unfinished and Wurlan finished plain weave fabrics laundered without agitation. All other percents should be different from the adjacent value with a 99.7% level of confidence.

Statistical Analysis of Clay and Oil Retention. Significantly more ($\alpha = .01$) clay was retained by plain weave fabrics than by twill weave fabrics (Table 3), but individual samples do not reveal a meaningful difference (Figure 3). The hypothesis that plain weave fabrics retain less clay than twill weave fabrics has been rejected for unfinished wool and the results were inconclusive for the Wurlan finished wool fabrics. The Wurlan finished plain and twill weave fabrics retained more ($\alpha = .001$) clay than the unfinished plain and

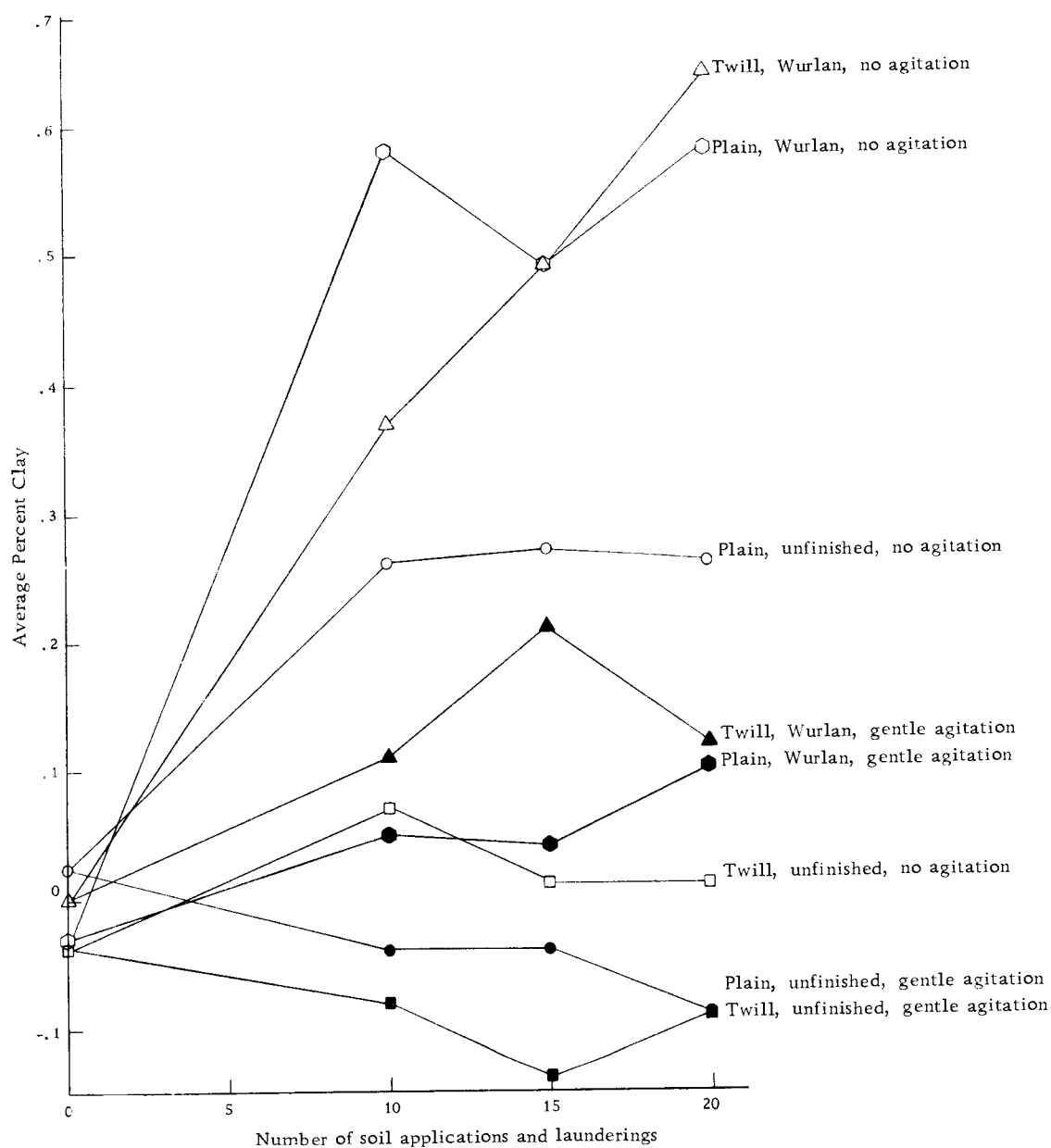


Figure 3. Clay content of soiled and laundered fabrics.

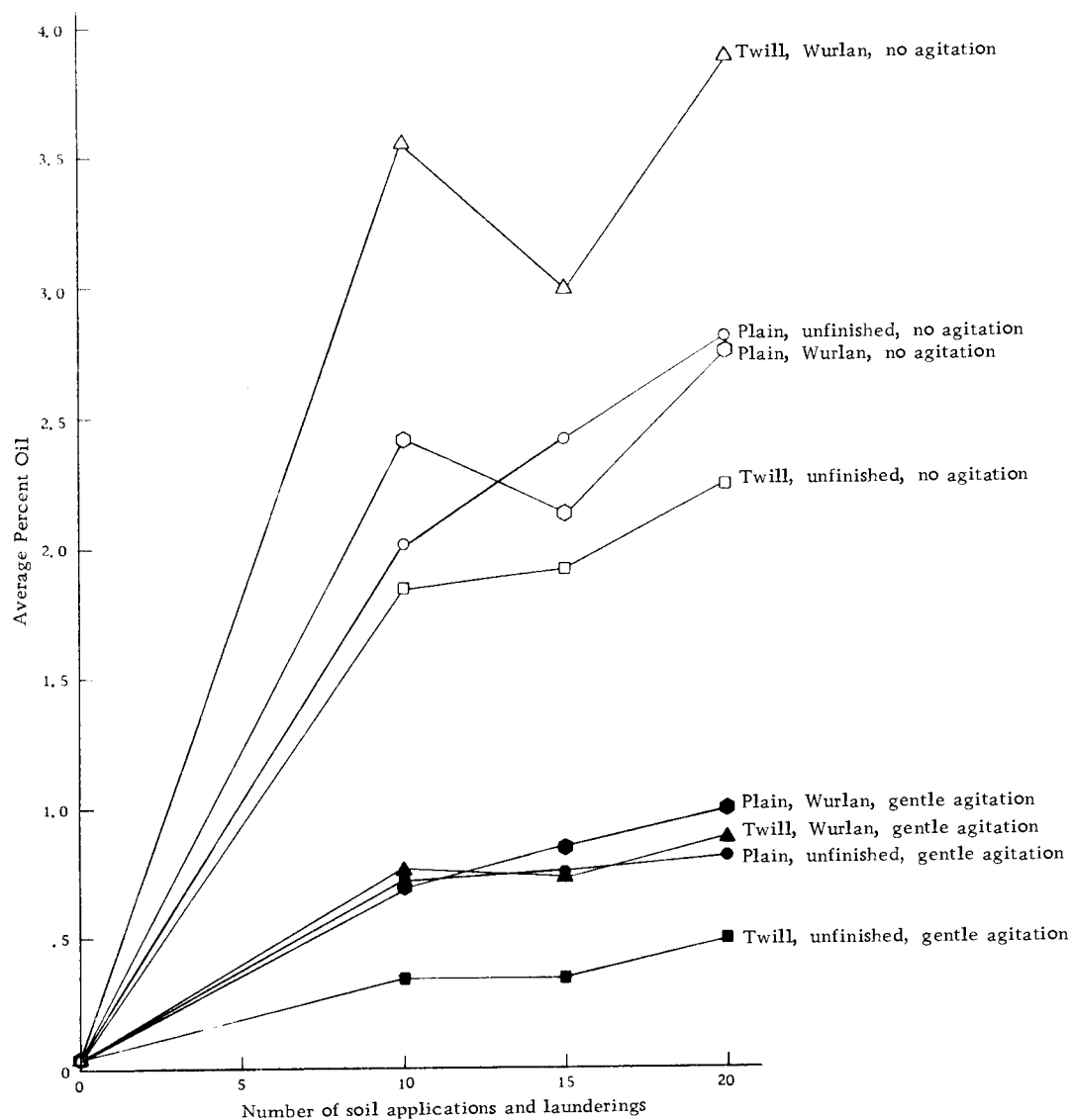


Figure 4. Oil content of soiled and laundered fabrics.

Table 3. Analyses of variance of percentage of clay and oil retained by laundered wool fabrics.

Source of variation	d.f.	Clay		Oil	
		Mean square	F value	Mean square	F value
Weave (W)	1	0.0666	11.58**	0.0165	0.16
Finish (F)	1	1.4120	245.63***	3.1375	29.66***
W × F	1	0.1096	19.06***	2.4531	23.19***
Agitation (A)	1	1.8744	326.05***	59.0241	557.96***
W × A	1	0.1198	20.84***	0.6593	6.23*
F × A	1	0.1716	29.85***	0.5707	5.39*
W × F × A	1	0.0041	0.71	0.8256	7.80**
Time (T)	2	0.0054	0.93	0.5279	4.99*
W × T	2	0.0059	1.03	0.0823	0.78
F × T	2	0.0177	3.07	0.0920	0.87
W × F × T	2	0.0182	3.16	0.0270	0.26
A × T	2	0.0073	1.27	0.0993	0.94
W × A × T	2	0.0040	0.70	0.0458	0.43
F × A × T	2	0.0072	1.26	0.1483	1.40
W × F × A × T	2	0.0170	2.96	0.0208	0.20
Error	48				
Total	71				

*** 0.1% level of significance

** 1.0% level of significance

* 5.0% level of significance

twill weave fabrics under the same laundering conditions resulting in acceptance of hypothesis 3. The similarity in clay retention of the Wurlan finished fabrics probably resulted from characteristics of that finish and/or similar dimensional changes.

The unfinished fabrics retained no clay when laundered with gentle agitation after 20 soilings and launderings, while the Wurlan finished fabrics retained a slight amount of clay leading to acceptance of hypothesis 4. Laundering without agitation resulted in significantly more ($\alpha = .001$) clay retention for all fabrics except the unfinished twill weave fabric which retained no detectable clay. However, all fabrics laundered both with and without agitation actually had little buildup of clay, with the highest level after 20 soil and laundry treatments being only 1/3 of the amount deposited at a single application. Wurlan finished twill weave fabrics laundered without agitation retained the most clay; 1/60 of the total amount of the clay that was applied. While there does appear to be greater retention of clay by the Wurlan finished fabrics laundered without agitation, it would not be a laundering problem since this fabric could be laundered with gentle agitation. The Wurlan finish may have an affinity for clay. It is also possible that the Wurlan stabilization which involves some spot welding, may create more nooks which can mechanically trap the clay so that there is a greater retention of clay by these fabrics.

While there was no significant buildup of clay over the period of this study, significantly more ($\alpha = .05$) oil was retained by all fabrics after 20 launderings than after ten, indicating that a slight amount of oil was retained at each laundering. It would seem that this accumulation of oil on wool fabrics might contribute to yellowing, particularly for white fabrics. However, yellowing was not detected in this study. The Wurlan finished fabric remained white and the greige unfinished fabrics became whiter after 20 launderings with gentle agitation, while those fabrics laundered without agitation were gray to brown due to the clay retained by these fabrics.

An analysis of variance indicated that significantly more oil ($\alpha = .001$) was retained by Wurlan finished than by unfinished wool fabrics resulting in acceptance of hypothesis 3. This effect was most pronounced for the twill weave, but the plain weave laundered with gentle agitation also retained slightly more oil (Figure 4). However, the unfinished plain weave fabric retained slightly more oil than the Wurlan finished plain weave when laundered without agitation.

Agitation appears to be a major factor in removal of oil as well as clay. No agitation resulted in significantly more ($\alpha = .001$) oil retention than gentle agitation (Figure 4) indicating acceptance of hypothesis 4. All fabrics retained two to four times more oil when not agitated than when gently agitated. Fabrics, except the

unfinished twill weave, laundered with gentle agitation, retained similar but significantly different amounts of oil. Possibly the unfinished twill weave retained slightly less oil because shrinkage reduced the surface area readily accessible for deposition. Laundering without agitation resulted in less oil retention (2.25%) for the unfinished twill weave fabrics than for the Wurlan finished twill weave fabrics (3.76%), again possibly due to shrinkage of the unfinished fabric which made deep penetration of oil applied more difficult. The Wurlan finish may have a greater affinity for oil, but if this is the case, it would be expected that both weaves would demonstrate this difference. There was a significant interaction ($\alpha = .01$) between weave, finish and agitation.

Figures 3 and 4 indicate that Wurlan finished fabrics retained small amounts of clay and relatively larger amounts of oil after 20 soil and laundry treatments. The design of this experiment does not permit determination of what influence, if any, oil retention had upon clay retention. It was noted that individual specimens were highly variable in the ratio of clay to oil at any given interval.

The unfinished fabrics laundered with gentle agitation had no detectable buildup of clay even though there was 0.50-0.85% buildup of oil. Laundering without agitation resulted in no further buildup of clay on unfinished fabrics after the tenth laundering while oil retention continued to increase. Therefore, oil retention by

unfinished fabrics clearly increased over time while there was little or no retention of clay. These results do not reveal any interaction between oil and clay on wool fabrics. Hodam (1965, p. 121) came to a similar conclusion after laundering artificially soiled wool yarns.

Redeposition of Clay and Oil onto Laundered but not Soiled Fabrics. There was no redeposition of clay during the 20 launderings under the conditions used in this study. Comparison of oil content of redeposition specimens over time (Figure 5) ranks the amounts of oil deposited on fabrics in similar order to the amounts retained by the same fabrics. Again, the unfinished twill weave fabric picked up more oil without agitation than with gentle agitation, and the Wurlan finished twill weave picked up more oil than the unfinished twill weave under both conditions. This again appears to indicate a soil and shrinkage interaction.

Richards (1966, p. 47, 54) found no redeposition of dibromostearic acid from Wurlan finished wool specimens soiled with 4.1% oil at each of ten applications. Over this period of time, 35% oil by weight of fabric was removed with low agitation and a temperature of $113 \pm 5^{\circ}\text{F}$. This was comparable to laundering with gentle agitation and 120°F . in this study where 2.0% oil by weight of the fabric was applied at each of 20 intervals and a total of 38% oil was removed during 20 launderings. Richards (p. 50, 61) observed that the bromine count rate decreased for the unsoiled Wurlan finished wool

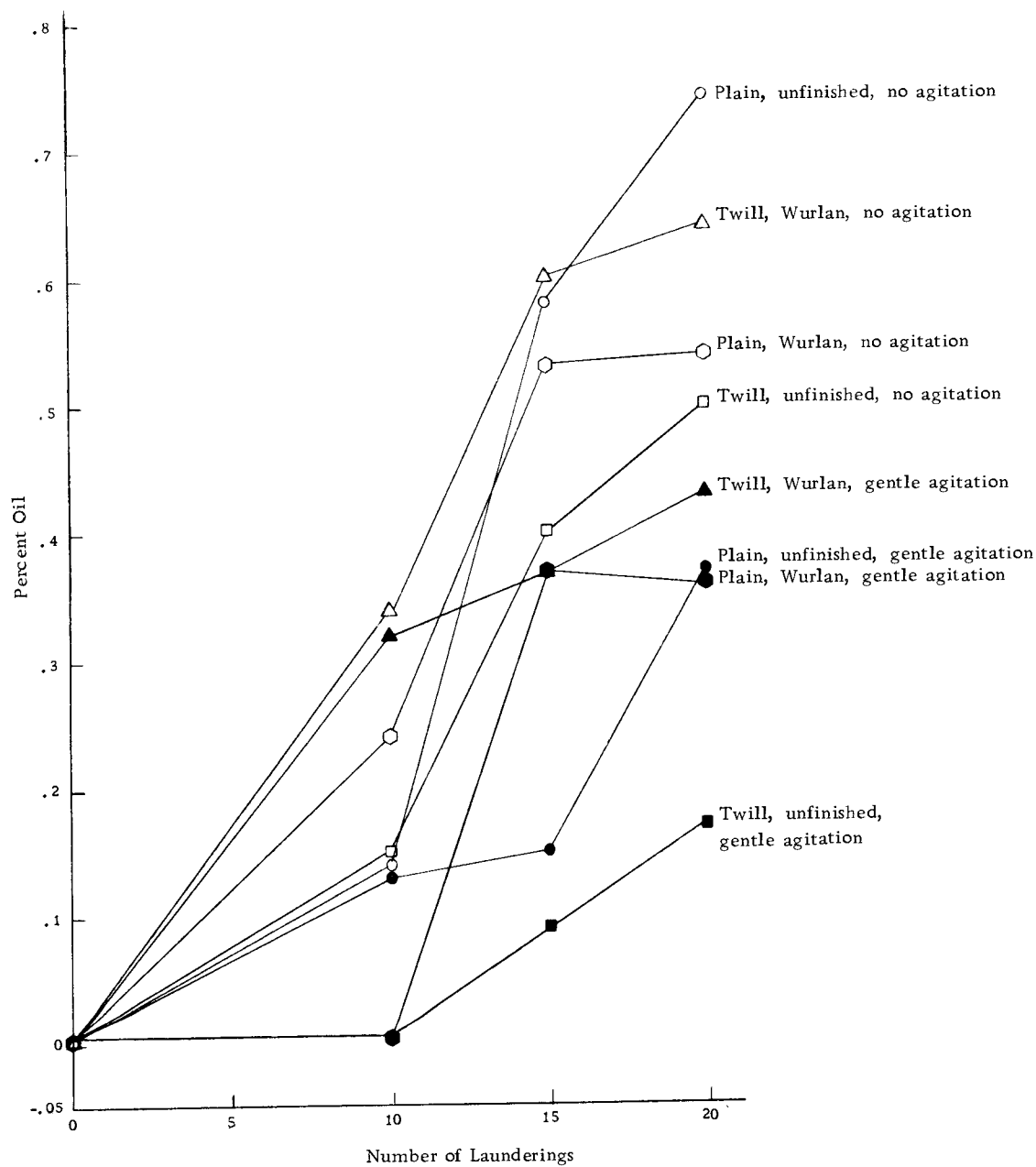


Figure 5. Oil content of single redeposition specimens.

fabrics and remained constant for the Wurlan finished mohair/wool fabrics as the tenth laundering approached. While the level of oil per laundry load which was available for redeposition was almost twice as much in her study, she found no redeposition. Possibly the standard anionic detergent used in this study was not as effective in prevention of redeposition as the cold-water nonionic detergent used by Richards. This reasoning is based on the previously noted efficiency of nonionic detergent in prevention of redeposition (Trowbridge, 1964). It is also possible that the quality of the water used may have affected the relative effectiveness of these detergents.

Dimensional Change and Breaking Strength of Wool
Fabrics Laundered with Standard Anionic and
Cold-Water, Nonionic Detergents

Dimensional Change

The percentage of warp and filling shrinkage caused by laundering these four wool fabrics was compared (Figures 6, 7, 8, 9, 10, 11, 12 and 13). The most obvious effect noted from these figures is that gentle agitation resulted in considerable shrinkage of unfinished plain and twill weave fabrics, but did not greatly affect the dimensions of Wurlan finished fabrics. The average shrinkage of unfinished wool fabrics after 20 launderings was 3.4% for the plain weave and 8.5% for the twill weave, with large variations between specimens depending upon the laundering conditions.

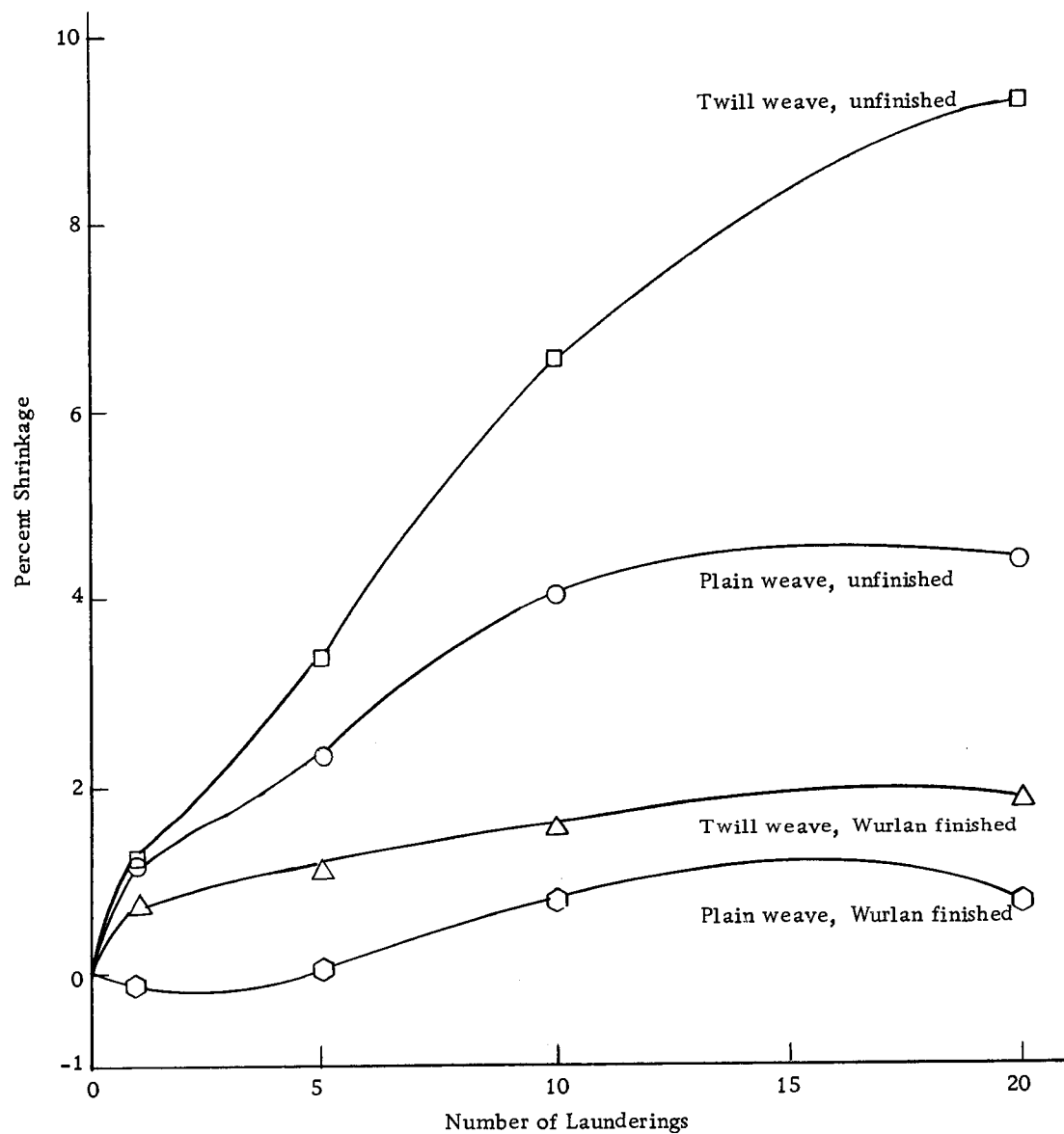


Figure 6. Percentage of warp shrinkage resulting from Cold Water "all" without agitation.

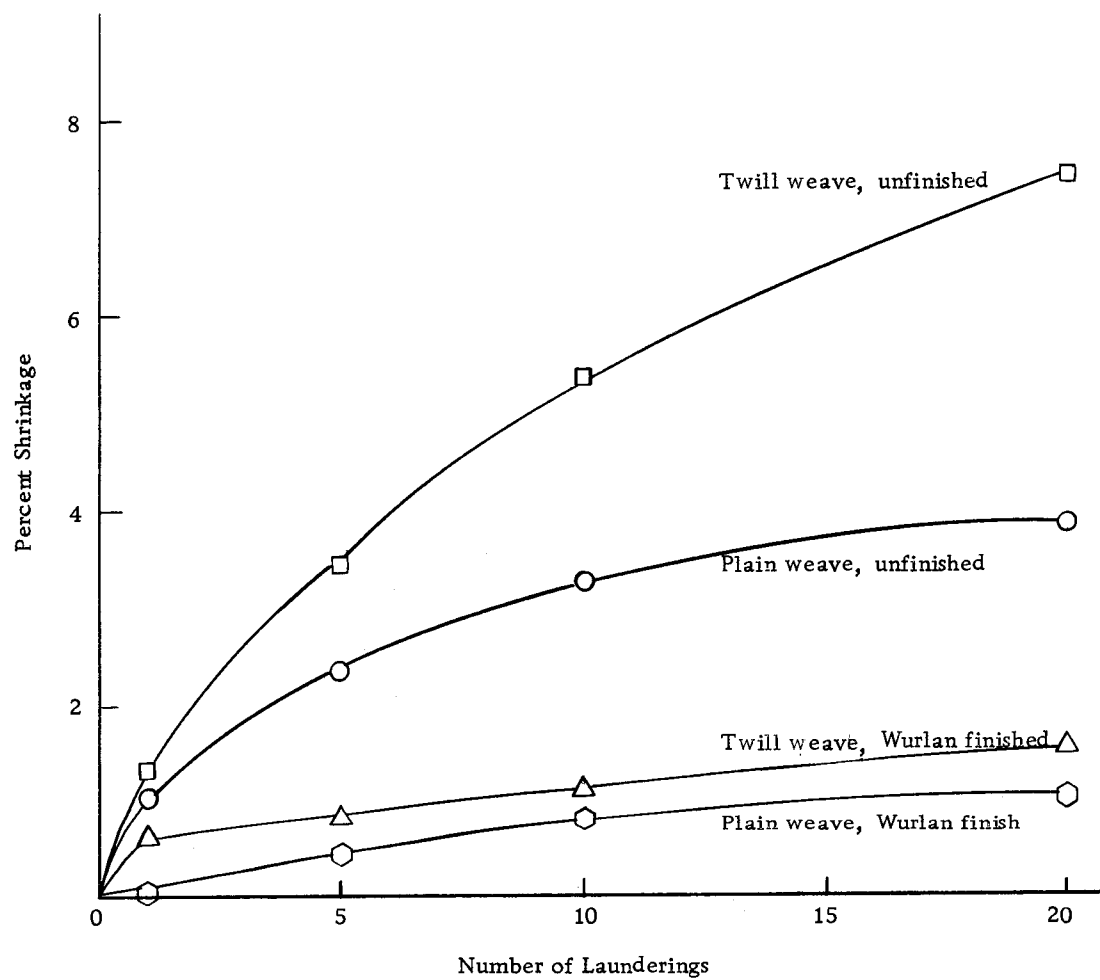


Figure 7. Percentage of warp shrinkage resulting from "Tide" without agitation.

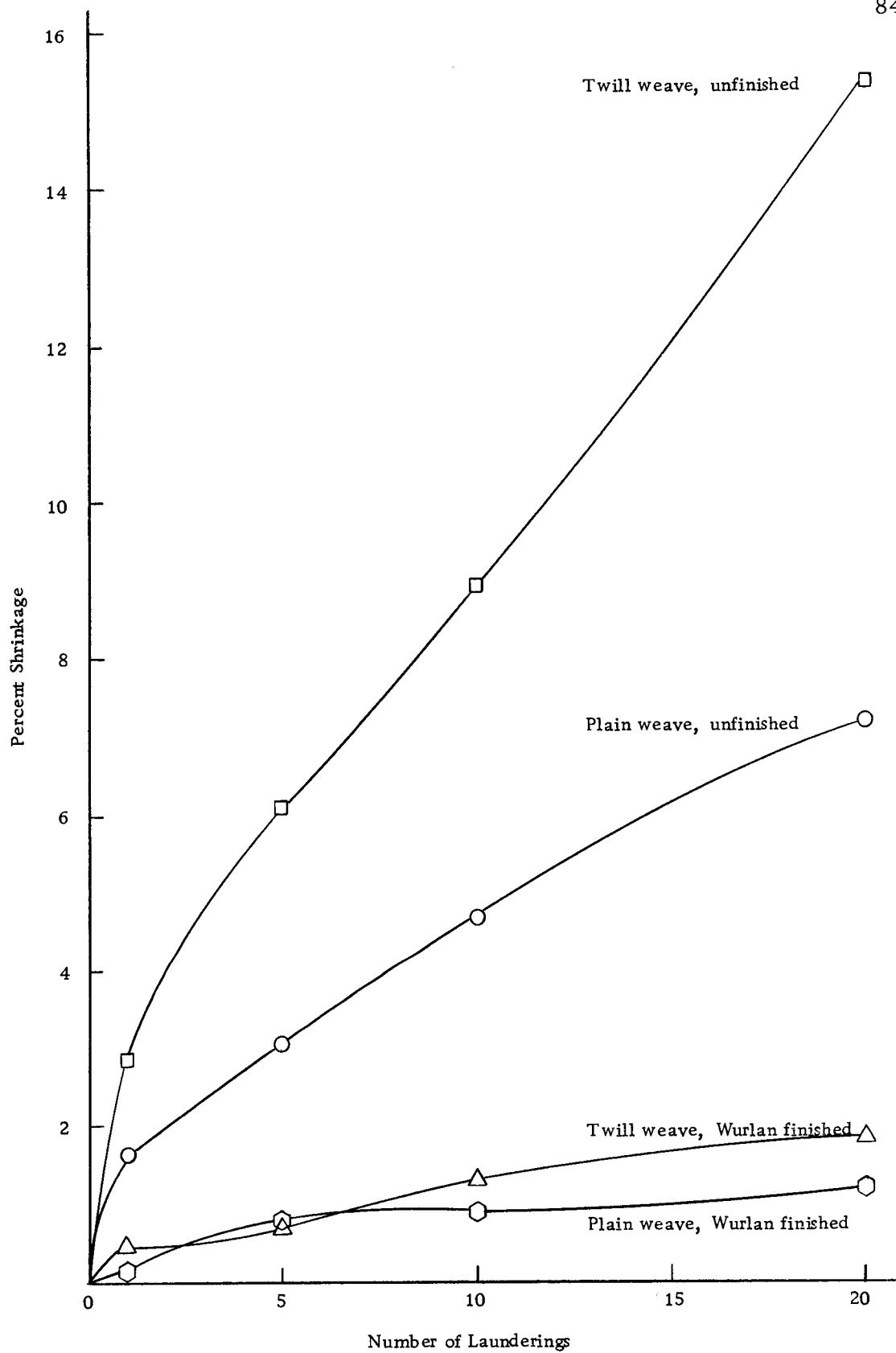


Figure 8. Percentage of warp shrinkage resulting from Cold Water "all" with gentle agitation.

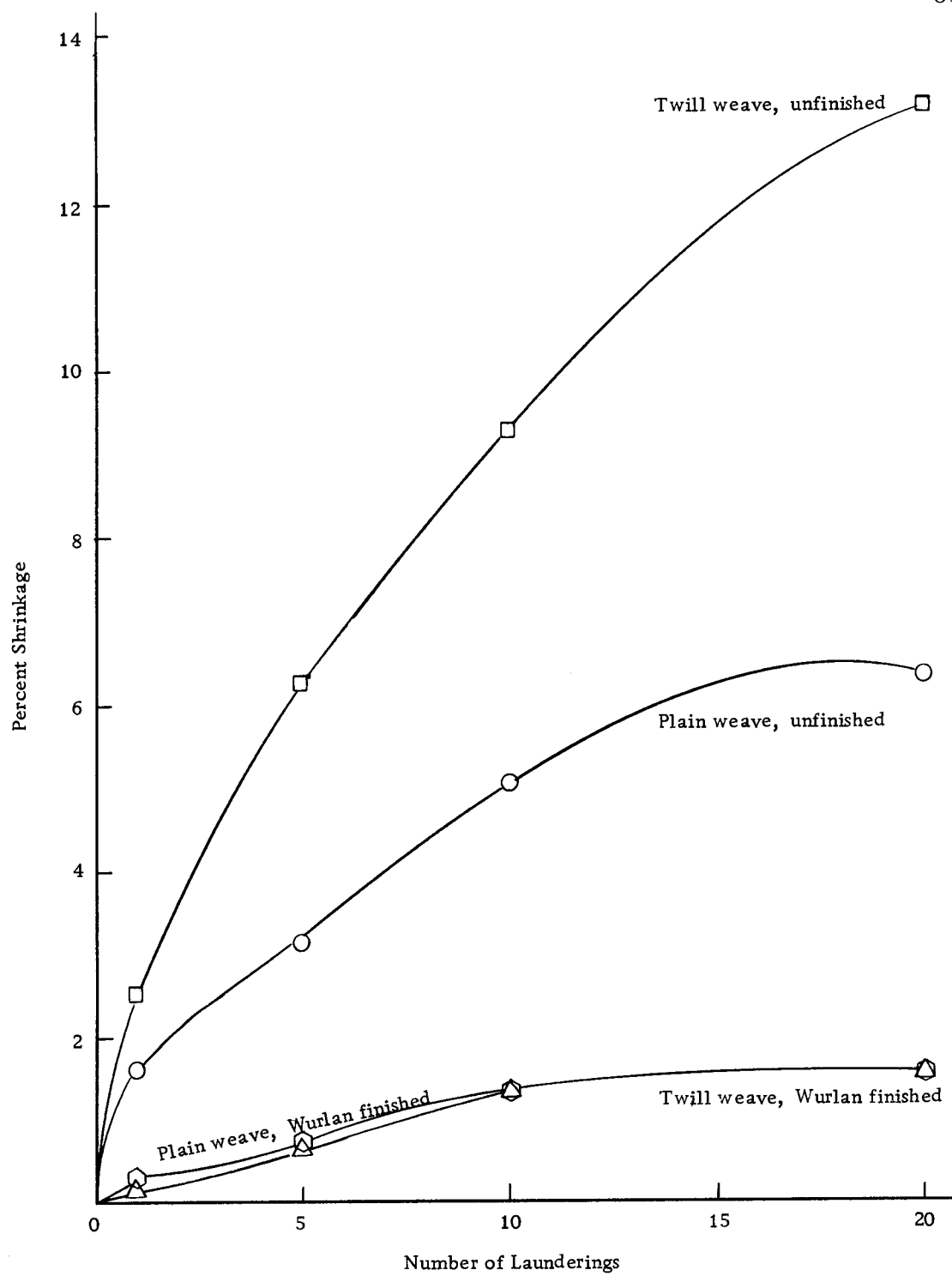


Figure 9. Percentage of warp shrinkage resulting from "Tide" with gentle agitation.

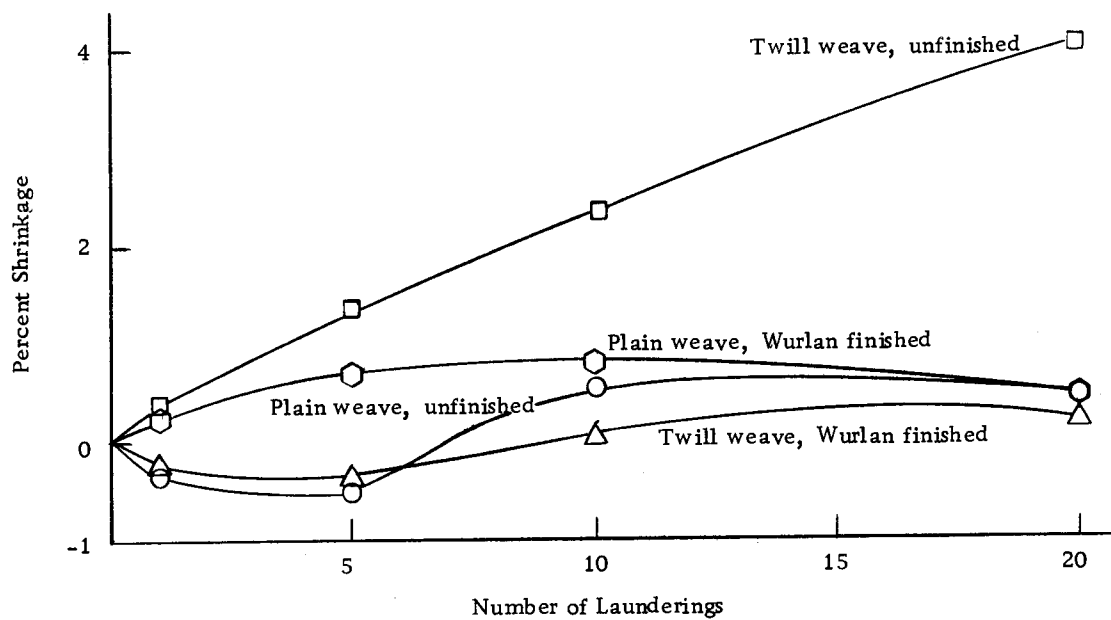


Figure 10. Percentage of filling shrinkage resulting from Cold Water "all" without agitation.

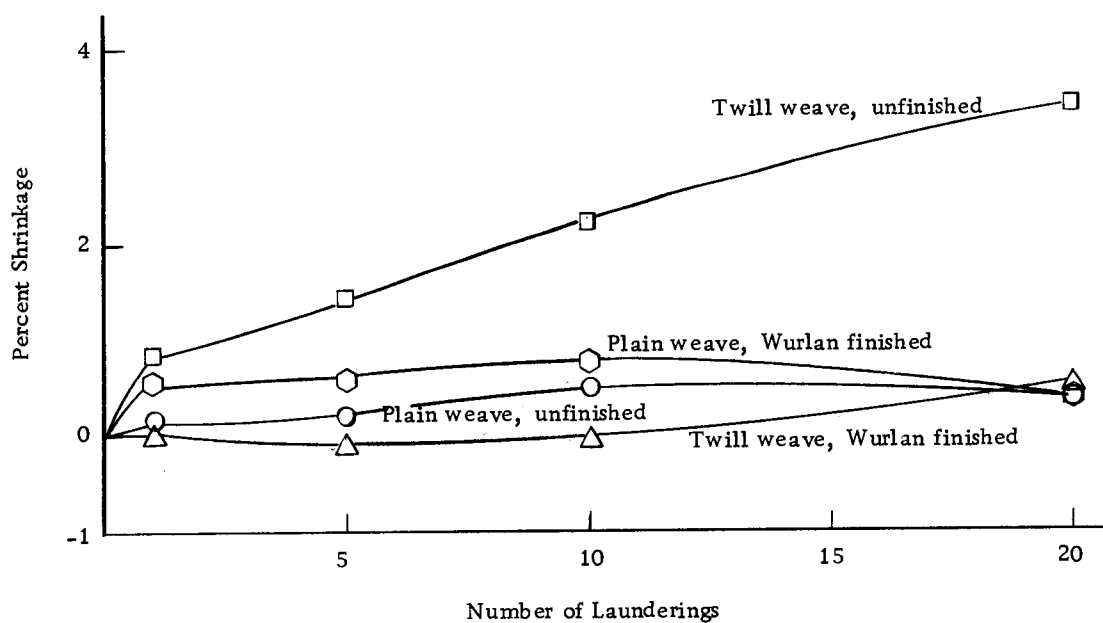


Figure 11. Percentage of filling shrinkage resulting from "Tide" without agitation.

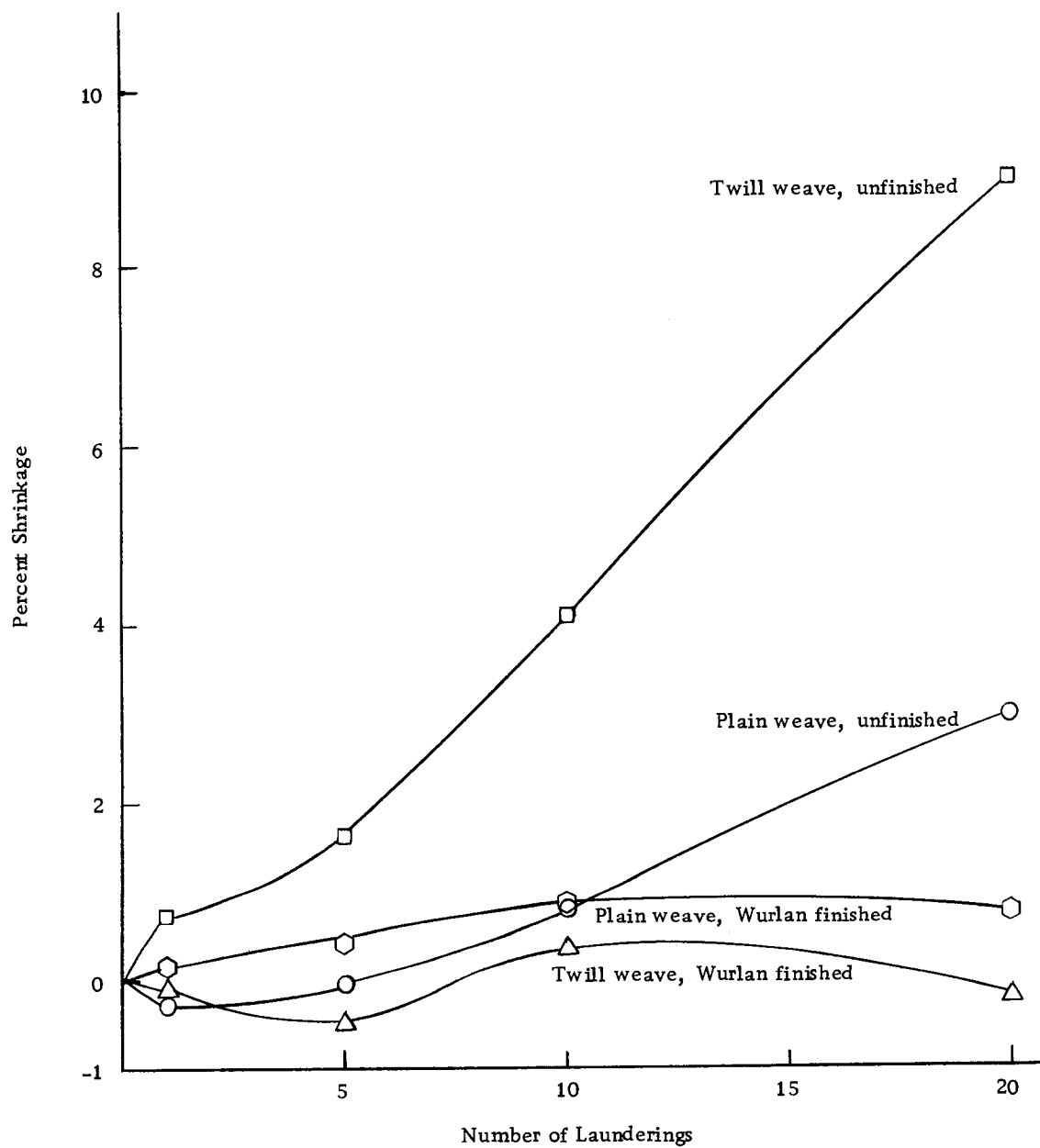


Figure 12. Percentage of filling shrinkage resulting from Cold Water "all" with gentle agitation.

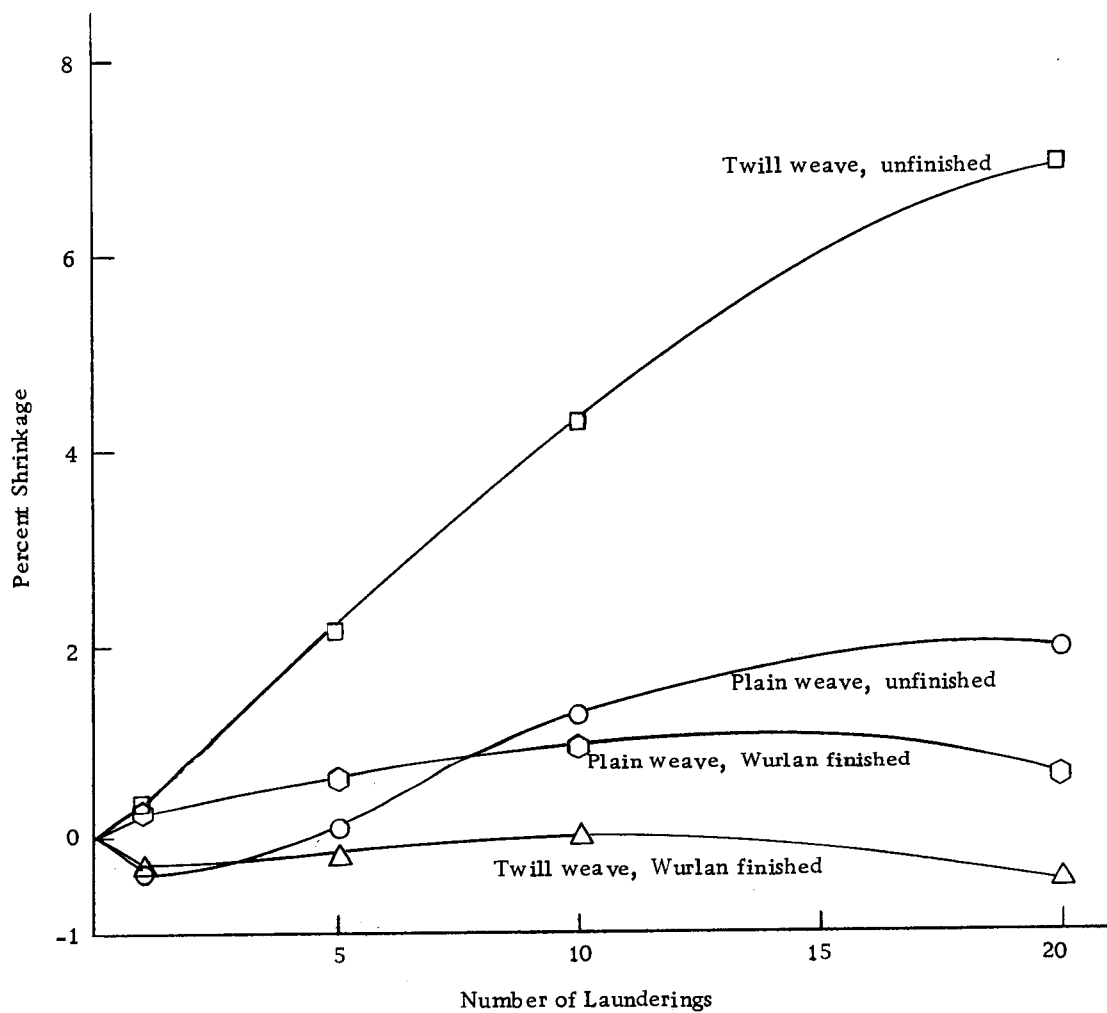


Figure 13. Percentage of filling shrinkage resulting from "Tide" with gentle agitation.

Under all conditions for all fabrics tested, the warp direction tended to shrink more and at a higher rate than the filling direction (Tables 4 and 5). In the filling direction, all unfinished fabrics shrank some while there was a tendency for Wurlan finished fabrics to relax slightly, particularly during the first ten launderings. However, this relaxation was always less than 0.5%.

The Wurlan finished fabrics all shrank less than 2% in both the warp and filling directions after 20 launderings. This excellent dimensional control substantiates early reports (Washable Woolens, 1964) that shrinkage of the "H₂O" (Wurlan) finished washable woolens by J. P. Stevens Co. would be under 3%. Contrary to early plant trials by Fong, Ash and Miller (1963), the shrinkage measurements also indicated that there was little difference between the two weaves finished with Wurlan. They found that a multicycle mild wash and tumble drying for five cycles resulted in greater shrinkage of Wurlan finished twill weave fabrics (3-4% warp and 1.7-3.3% filling) than of plain weave fabrics (1.2-2.3% warp and 0.8-2.5% filling). Possibly the detergent, the washer, or the tumble dryer contributed to the difference in shrinkage between weaves and to the greater overall shrinkage that they observed.

Statistical Analysis of Shrinkage

Both the weave and the finish were significant variables

Table 4. Average percentage of shrinkage of wool fabrics laundered with cold-water, nonionic detergent.

Fabric	Laundering							
	1		5		10		20	
	Warp	Filling	Warp	Filling	Warp	Filling	Warp	Filling
<u>Gentle agitation</u>								
Unfinished, plain weave	1.64	+0.31	3.04	+0.04	4.70	0.80	7.19	2.97
Wurlan, plain weave	0.16	0.13	0.77	0.40	0.90	0.82	1.19	0.77
Unfinished, twill weave	2.87	0.73	6.11	1.62	8.92	4.09	15.32	8.96
Wurlan, twill weave	0.50	+0.11	0.73	+0.48	1.31	0.36	1.83	+0.19
<u>No agitation</u>								
Unfinished, plain weave	1.12	+0.34	2.33	+0.54	4.03	0.52	4.38	0.46
Wurlan, plain weave	+0.14	0.22	0.03	0.70	0.74	0.79	0.72	0.47
Unfinished, twill weave	1.24	0.37	3.39	1.37	6.54	2.33	8.78	4.05
Wurlan, twill weave	0.74	+0.21	1.10	+0.38	0.57	0.03	1.82	0.21

Table 5. Average percentage of shrinkage of wool fabrics laundered with standard, anionic detergent.

Fabric	Laundering							
	1		5		10		20	
	Warp	Filling	Warp	Filling	Warp	Filling	Warp	Filling
<u>Gentle agitation</u>								
Unfinished, plain weave	1.61	+0.38	3.13	0.10	5.04	1.27	6.35	1.96
Wurlan, plain weave	0.28	0.27	0.71	0.60	1.32	0.97	1.57	0.65
Unfinished, twill weave	2.55	0.37	6.28	2.14	9.27	4.30	13.12	6.91
Wurlan, twill weave	0.15	+0.33	0.67	+0.18	1.31	0.01	1.56	+0.43
<u>No agitation</u>								
Unfinished, plain weave	1.04	0.16	2.34	0.19	3.25	0.49	3.86	0.38
Wurlan, plain weave	0.01	0.53	0.43	0.59	0.81	0.75	1.07	0.38
Unfinished, twill weave	1.32	0.82	3.44	1.42	5.34	2.20	7.42	3.39
Wurlan, twill weave	0.68	+0.02	0.82	+0.13	1.12	+0.06	1.59	0.51

($\alpha = .001$) in both warp and filling shrinkage of these unfinished and Wurlan finished wool fabrics (Table 6). The plain weave fabrics shrank less than twill weave fabrics so hypothesis 5 has been accepted. In the warp direction, shrinkage of Wurlan finished plain weave fabrics was less than or equal to the twill weave. However, laundering with no agitation resulted in slight growth of the twill weave in the filling direction. Therefore, the overall filling shrinkage of Wurlan finished twill weave fabric was less than the shrinkage of plain weave fabric. The Wurlan finished twill weave fabric achieved the greatest shrinkage resistance over its unfinished counterpart. Warp shrinkage was significantly greater ($\alpha = .001$) for the unfinished than for the Wurlan finished plain and twill weave fabrics. The unfinished twill weave also shrank significantly more ($\alpha = .001$) in width than the Wurlan finished twill, but there was little difference between the minimal filling shrinkage of unfinished and Wurlan finished plain weave fabrics. Hypothesis 6 has been accepted on the basis of these results.

The shrinkage figures indicate that gentle agitation resulted in significantly more ($\alpha = .001$) warp shrinkage. While a similar trend in filling shrinkage, resulting from agitation, can be seen in Figures 10, 11, 12, and 13, the difference between gentle and no agitation was not significant. Thus hypothesis 7 was confirmed. The weave and agitation interaction was significant ($\alpha = .10$) in both directions

Table 6. Analysis of variance of warp and filling shrinkage of wool fabrics.

Source of variation	d.f.	F ratio for warp	F ratio for fill	Source of variation	d.f.	F ratio for warp	F ratio for fill
Weave (W)	1	120.74****	35.38****	Time (T)	3	611.43****	19.99***
Finish (F)	1	720.93****	81.68****	Error (T)	6		
W x F	1	34.68****	192.93****	W x T	3	14.28****	0.97
Detergent (D)	1	1.75	0.35	F x T	3	99.11****	0.54
W x D	1	2.65	2.81	W x F x T	3	0.48	31.36****
F x D	1	1.25	0.01	D x T	3	0.67	3.09**
W x F x D	1	0.31	0.00	W x D x T	3	0.75	1.46
Agitation (A)	1	72.79****	3.25	F x D x T	3	0.44	0.36
W x A	1	3.64*	3.87*	W x F x D x T	3	0.52	0.20
F x A	1	71.35****	9.58***	A x T	3	12.65****	6.61****
W x F x A	1	30.02****	3.58	W x A x T	3	0.08	0.41
D x A	1	0.14	7.96***	F x A x T	3	17.29****	0.88
W x D x A	1	0.53	0.51	W x F x A x T	3	11.57****	0.13
F x D x A	1	0.02	2.11	D x A x T	3	2.03	8.62****
W x F x D x A	1	0.08	0.00	W x D x A x T	3	0.92	0.80
Error	32			F x D x A x T	3	0.32	1.62
Total	191			W x F x D x A x T	3	0.25	0.27
				Error	90		

*10.0% level of significance.

**5.0% level of significance.

***1.0% level of significance.

****0.1% level of significance.

due to greater shrinkage of the unfinished twill weave particularly with gentle agitation. The unfinished twill weave fabric was most affected by gentle agitation with a warp shrinkage of five in./yd. as opposed to 2.5 in./yd. for unfinished plain weave fabrics. There was a significant relationship between finish and agitation in both directions, with the unfinished fabrics of both weaves shrinking more ($\alpha = .001$) with gentle agitation than without agitation. The Wurlan finish resulted in excellent resistance ($\alpha = .001$) to shrinkage in both the warp and filling directions during laundering with gentle agitation.

Hypothesis 8 was rejected because there does not appear to be a real difference in dimensional change of fabrics laundered with anionic and nonionic detergents. The only significant interactions noted for detergent were in the filling direction for detergent and agitation, detergent and time, and detergent, agitation and time. A combination of gentle agitation and anionic detergent resulted in significantly more ($\alpha = .01$) filling shrinkage than cold-water nonionic detergent without agitation. Gentle agitation with nonionic detergent resulted in more shrinkage than anionic detergent without agitation. This indicates that the dominant effect on felting shrinkage was agitation rather than detergent. The interaction of detergent and time in the filling shrinkage reveals that over time (20 launderings) the cold-water nonionic detergent produced significantly

more ($\alpha = .05$) filling shrinkage than the standard anionic detergent. However, it should be noted that filling shrinkage was not as excessive as warp shrinkage, where the two detergents under the same conditions caused similar shrinkage.

The rate of shrinkage of these wool fabrics was significantly greater during early launderings. This effect was most prominent for unfinished wool fabrics. The warp direction of all fabrics had a more significant decrease ($\alpha = .001$) in the rate of shrinkage than the filling direction ($\alpha = .01$). The most warp shrinkage per laundering occurred at the first laundering with continued shrinkage at a lower rate for the 19 remaining launderings. Greater warp shrinkage ($\alpha = .001$) of twill weave fabrics than of plain weave fabrics occurred during early launderings. Wurlan finished fabrics reached a plateau of dimensional change by about the tenth laundering and there was little change in dimensions between the tenth and 20th launderings. The unfinished fabrics were shrinking almost as much per laundering at the 20th laundering as the finished fabrics did at the first laundering.

The relationship between agitation and time is significant ($\alpha = .001$) for both warp and filling shrinkage of these wool fabrics. Except for the warp direction of the Wurlan finished twill weave, all fabrics shrank more over time with gentle agitation than without agitation.

There were several significant multiple interactions. Finish, agitation, and time were significantly related ($\alpha = .001$) in the warp direction. A four factor interaction between weave, finish, agitation and time ($\alpha = .001$) in the warp direction was also found.

Breaking Strength

The average warp grab breaking strength of these four wool fabrics after 20 launderings has been compared to unlaundered controls (Table 7). Comparison of these unlaundered controls indicated that the Wurlan finish resulted in either greater breaking strength (plain weave) or equal breaking strength (twill weave). This verifies results of cut strip breaking strength tests of unlaundered fabrics by Whitfield, Miller and Wasley (1961) and Fong et al. (1962, 1963).

Statistical Analysis of Breaking Strength

The analysis of variance of warp grab breaking strength (Table 8) indicated that fabric, finish, and treatment, as well as all of their interactions were significant variables. The fabrics and finishes were both significantly different ($\alpha = .001$) because the breaking strength of the unfinished plain weave fabric was consistently lower than that of the other three fabrics. Therefore, hypothesis 9 has been accepted for the plain weave fabrics but rejected for the twill weave

Table 7. Average warp grab breaking strength of wool fabrics.*

Treatment	Fabric			
	Plain** unfinished	Plain Wurlan	Twill*** unfinished	Twill Wurlan
Control unlaundered	24.3	30.5	31.3	32.0
Cold Water "all, " no agitation ⁺	24.5	34.1	32.5	34.7
Cold Water "all, " gentle agitation ⁺	24.5	31.4	36.4	35.5
"Tide, " no agitation ⁺	23.3	29.7	31.8	33.5
"Tide, " gentle agitation ⁺	22.7	31.3	34.2	33.7

* Averages of five successful breaks expressed in lb/sq.in.

** Plain weave

*** Twill weave

+ Laundered for five minutes at 120°F.

Table 8. Analysis of variance of warp grab breaking strength of wool fabrics.

Source of variation	d.f.	Sum of squares	Mean squares	F ratio
Weave (W)	1	876	876	376**
Finish (F)	1	420	420	180**
Treatment (T)	4	94	23.50	10**
W × F	1	296	296	127**
W × T	4	55	13.75	5.9**
F × T	4	26	6.50	2.78*
W × F × T	4	298	74.50	31**
Error	80	187	2.33	
Total	99	1969		

** 0.1% level of significance

* 5% level of significance

fabrics. The treatments resulted in a significant difference ($\alpha = .001$) because some laundering conditions, particularly for the twill weave, resulted in higher breaking strength than for the unlaundered controls. This unexpected result caused the rejection of hypothesis 10. The interaction between the fabric and treatment is more significant ($\alpha = .001$) than the interaction between finish and treatment ($\alpha = .05$). This reflects the fact that the unfinished and Wurlan finished plain weave fabrics produced more consistent breaking strength measurements regardless of the treatment. This difference in breaking strength might be explained by the physical structure of the weave and also by the greater and more variable shrinkage of the unfinished twill weave fabric which resulted in a more compact structure.

To determine more specifically the effect of the Wurlan finish upon breaking strength under given conditions, some least significant differences were performed (Appendix C). These analyses indicated that finishing of plain weave fabrics with Wurlan resulted in significantly higher ($\alpha = .01$) breaking strength. Unfinished and Wurlan finished twill weave fabrics were not significantly different except when laundered with Cold Water "all" without agitation where the Wurlan finished twill was stronger than the unfinished twill weave ($\alpha = .05$).

Wurlan finished plain weave fabric was significantly stronger ($\alpha = .01$) when laundered with Cold Water "all" without agitation

than under all other laundering conditions ($\alpha = .05$). Wurlan finished twill weave fabrics laundered with Cold Water "all" with either gentle or no agitation also had a significantly higher ($\alpha = .05$) breaking strength than the unlaundered control. The reason for the increase in breaking strength is unknown but does not appear to be related to shrinkage. Table 7 gives some indication that breaking strengths of all fabrics laundered with the nonionic detergent tend to be higher. The unfinished twill weave fabrics laundered with both Cold Water "all" and "Tide" at gentle agitation had a significantly higher ($\alpha = .01$) breaking strength than the unlaundered control specimens. This increase may be due to shrinkage. A similar effect was noted in a study of wool blankets (Petzel et al., 1961).

It was hypothesized that the breaking strength and dimensional change of the Wurlan finished fabrics after 20 launderings would reflect the permanency of the finish for that length of time. It can be seen that there was no increase in the rate of shrinkage of the Wurlan finished specimens; a factor that would seem to indicate that the finish has not been removed sufficiently to reduce the performance during these 20 launderings. The breaking strength tests also indicated that any possible loss of finish was not accompanied by a decrease in tensile strength, particularly for the plain weave fabrics where breaking strengths of the unlaundered Wurlan finished specimens were higher than the unfinished plain weave specimens.

Based on these results, hypothesis 10 must be rejected for this period of 20 launderings.

Results of Selected Ravelled Strip Breaking Strength Tests

To determine if shrinkage of the unfinished twill weave fabric caused increased warp grab breaking strength after laundering, warp ravelled strip breaking strength tests were performed on two specimens each of unlaundered fabrics and fabrics laundered with Tide using gentle agitation. No further tests were made due to lack of fabric. These tests (Appendix D) indicate that the Wurlan finish increased the strength per yarn of both the plain and twill weave fabrics. Contrary to grab breaking strength tests (p. 96), the ravelled strip tests seem to support the hypothesis that the twill weave fabric would gain strength when finished with Wurlan.

Laundering with Tide at gentle agitation caused a decrease in the warp breaking strength per yarn for all fabrics in contrast to the frequent increase in fabric strength noted with the grab test (p. 99). Shrinkage of the laundered fabrics apparently resulted in higher warp grab breaking strength. The decrease in ravelled strip breaking strengths of Wurlan finished fabrics does not necessarily indicate removal of the finish. These breaking strength values even after laundering are higher than the initial values for the unfinished fabrics.

RECOMMENDATIONS FOR FURTHER STUDY

X-ray fluorescent spectroscopy is a relatively rapid and easy method for quantitative detection of artificial soil retained by fabrics. However, a technique for the preparation of clay standards should be perfected to increase the accuracy of replication of these standards. This might involve either preparation of a large number of standards, or development of an alternative method of preparation of these standards.

It would be quite feasible to determine the relative amounts of particulate soil retained by naturally soiled fabrics with X-ray fluorescent spectroscopy. This could be done by counting several common elements such as aluminum, silicon, iron and manganese. If sufficient particulate soil were retained by fabrics for a quantitative determination, the clay minerals present could be identified by X-ray diffraction of the ash. These clay minerals then could be deposited on fabrics as standards for determination of the amounts of these minerals retained by the naturally soiled fabrics.

The detection of a natural oil by X-ray fluorescent spectroscopy would be considerably more difficult since body oil is complex and contains no element which can be excited by X-ray fluorescent spectroscopy. Therefore, an element such as bromine would have to be introduced in some way. Bromine would be expected to add

readily across the double bonds of unsaturated molecules such as oleic acid, triolein, and possibly cholesterol. However, addition of bromine to the oil-fabric complex is likely to be messy and would probably result in a relatively high count rate for fabrics which actually have no oil. It might be possible to extract the oil from the fabric, brominate the unsaturated oils, and filter the oil to separate it from excess bromine. This separation could be achieved by use of a material which differentiates molecular size by retaining small molecules or atoms and allowing the larger molecules to pass through. This oil could then be excited by X-rays and compared to standard amounts of oil which have also been extracted from naturally soiled fabrics to give a quantitative estimate of oil retention.

There is still some doubt about the influence, if any, which increased oil content has upon the retention of particulate soil. Such a basic research problem could be readily investigated with X-ray fluorescent spectroscopy.

X-ray fluorescent spectroscopy has been used to detect soil retained by wool and mohair/wool (Richards, Morris and Arkley, 1968) after laundering. It should be possible to detect residual soil of fabrics containing other fibers.

Electron micrographs could provide further insight into the greater retention of clay and oil by the Wurlan finished fabrics. The new scanning electron microscope might be able to determine

the location and depth of the finish more accurately, and consequently the relation of this finish to the clay and oil retained.

It would be helpful to compare the amount of oil picked up by wool fabrics after launderings to determine if felted fabrics do pick up less oil. If felting did not appear to affect the amount of oil deposition, would it affect the location of that oil so that it would be more easily removed during launderings? While significantly more oil was retained by Wurlan finished fabrics than by unfinished wool fabrics, the difference between the plain weave fabrics was small. It would be desirable to use a larger sample of Wurlan finished fabrics to determine if this finish actually does have a greater affinity for oil.

Ravelled strip breaking strength tests on laundered Wurlan finished and unfinished wool would help to explain the strength which might be expected if there were no shrinkage, since the strength per yarn would be calculated. This test might reveal a greater difference between the unfinished and Wurlan finished fabrics after laundering.

SUMMARY AND CONCLUSIONS

The objectives of this study were (1) to develop a combination of soiling, laundering, and soil detection techniques which would result in an accurate and reasonably rapid test of the relative effectiveness of the laundering conditions used, (2) to develop a method of quantitative X-ray fluorescent spectroscopy of artificial soil retained by fabrics, (3) to compare the soil retention of Wurlan finished plain and twill weave wool fabrics to unfinished wool fabrics, and (4) to compare the shrinkage and breaking strength of unfinished and Wurlan finished wool fabrics after 20 launderings as an indication of the durability of this finish.

These fabrics were artificially soiled by immersion in a mixed oil (50% dibromostearic acid) suspended in perchlorethylene, dried, and then single specimens were tumbled with dry Spinks Bandy Black Clay. The relative ease of removal of this artificial soil from unfinished and Wurlan finished plain and twill weave fabrics was evaluated by X-ray fluorescent spectroscopy of the elements bromine (oil) and aluminum (clay) after laundering these fabrics at 120°F. for five minutes with a standard anionic detergent using either gentle or no agitation. The percentages of clay and oil retained by these fabrics were determined by comparison to count rates of fabric containing known percentages of clay and oil.

The method of application of oil to these wool fabrics by immersion was reproducible and relatively easy, but a method to simulate the natural application of body oil would be desirable. The dry application of clay to these wool fabrics was appropriate. However, a reduction of time involved in application of the clay and improvement of replication accuracy would have some merit.

Quantitative detection of soil retained by wool fabrics using X-ray fluorescent spectroscopy has been developed by this study. With some improvement in the preparation of standards, this method would be a desirable alternative to extraction and ashing, color difference, or radioactive tracer methods. The main advantages of this method are speed and accuracy. The preparation and counting of specimens is faster than extraction and ashing of an equal number of specimens, requiring only 20-30 minutes to analyze a single specimen for the percentages of residual oil and clay. This study indicated that it is possible to count elements such as aluminum and silicon retained by fabrics, even though these elements are at the lower limits of detection with the equipment available today.

Based on the results of this study, hypotheses 1, 3, 4, 5, 6, and 7 have been accepted while hypotheses 8 and 10 were rejected. Hypothesis 2 was rejected for the unfinished wool fabrics but inconclusive results were obtained for the Wurlan finished wool fabrics. Hypothesis 9 has been confirmed for the plain weave fabrics but not

for the twill weave fabrics.

Agitation was the most important factor affecting retention of clay and oil by the wool fabrics used in this study. Significantly less clay (little or none) and oil (two to four times less) were retained by fabrics laundered with gentle agitation than fabrics laundered without agitation.

The hypothesis that plain weave fabrics retain less soil than twill weave fabrics has been rejected for unfinished fabrics but evidence was inconclusive for the Wurlan finished fabrics. The variations in oil content appeared to be more related to the finish than to the weave, but the plain weave fabrics seemed to retain as much, if not more oil, than the twill weave fabrics.

There was not a significant buildup of clay on these wool fabrics after 20 soilings and launderings. However, the two Wurlan finished fabrics retained a similar amount of clay which was greater than that retained by unfinished fabrics. Possibly the finish, which involves some spot welding to restrict fiber mobility, creates a structure in which some clay can be mechanically trapped. However, the finish itself may simply have an affinity for clay.

Oil did build up on all of the fabrics over the period of this study. While there was little difference in oil retained by the unfinished and Wurlan finished plain weave fabrics, the Wurlan finished twill weave fabric retained considerably more oil than the

unfinished twill weave fabric. This difference may result from an affinity of the oil for the finish, but might also be a reflection of the greater shrinkage which made deep penetration of oil applied to the unfinished twill weave fabric more difficult. It is also possible that the oil content of these two fabrics is not as different as this analysis indicates since the difference in physical structure of these fabrics could affect the absorption and scattering of secondary X-rays.

This study verifies observations that gentle agitation can cause considerable shrinkage of unfinished plain and twill weave wool fabrics. These fabrics also shrank 4-10% when laundered without agitation. In contrast, shrinkage of the Wurlan finished wool fabrics was less than 2% in both the warp and filling directions after 20 launderings with both gentle and no agitation. This Wurlan finish was particularly effective on the twill weave fabric. Laundering with either standard anionic detergent or cold-water nonionic detergent resulted in similar shrinkage.

Significantly more warp shrinkage occurred during early launderings, particularly for the unfinished fabrics. The dimensions of the Wurlan finished fabrics were stable after the tenth laundering, while the unfinished fabrics were still shrinking at the 20th laundering.

The Wurlan finish increased the warp grab breaking strength

of the plain weave fabric but did not affect the breaking strength of the twill weave fabric. Laundering of unfinished twill weave specimens at gentle agitation with both detergents resulted in a significantly higher breaking strength than for the unlaundered specimens. This increased strength may have been caused by excessive felting shrinkage which resulted in a more compact fabric. The breaking strength of the Wurlan finished fabrics after 20 launderings was either greater than or equal to the unlaundered breaking strength. The results of both breaking strength and shrinkage tests seem to indicate that the Wurlan finish was effective for the length of this study.

Conclusions

1. The artificial application of oil to these wool fabrics was easier and more reproducible than the dry application of clay. However, the oil application by liquid immersion was not as realistic as the dry application of clay by tumbling.

2. X-ray fluorescent spectroscopy is a promising quantitative technique for detection of artificial soil retained by fabrics. The main advantages of this method are accuracy and the speed and the relative ease of preparation of specimens for analysis at a moderate cost.

3. These Wurlan finished fabrics retained slightly more clay

and oil than did the unfinished fabrics. Variations in weave and finish both affected the accumulation of oil with the greatest amount of oil being retained by the Wurlan finished twill weave fabrics. Gentle agitation removed most of the clay from these wool fabrics and two to four times more oil than no agitation. There was an undesirable accumulation of oil and a small amount of clay when these wool fabrics were laundered without agitation.

4. The Wurlan finished wool fabrics used in this study were quite resistant to felting shrinkage when laundered 20 times with both gentle and no agitation. A shrinkage of less than 2% for these fabrics was in sharp contrast to unfinished fabric shrinkage of as much as 16% with gentle agitation and 9% without agitation.

5. The excellent dimensional stability of these Wurlan finished wool fabrics permits laundering with gentle agitation and a standard synthetic detergent in moderately hot water (120°F.). These conditions result in less retention of soil than laundering of unfinished wool without agitation.

6. The warp grab breaking strength of the Wurlan finished plain weave fabric was slightly higher than that of the unfinished plain weave fabric, but the finish did not increase the warp grab breaking strength of the twill weave fabric. However, limited ravelled strip tests indicated that the Wurlan finish increased the strength per warp yarn of both fabrics. The warp grab breaking strength of the Wurlan

finished fabrics after 20 launderings was equal to or greater than that of the unlaundered Wurlan finished fabrics. The warp breaking strength per yarn of these fabrics, as determined by ravelled strip tests of selected samples, decreased slightly after 20 launderings.

7. Shrinkage and breaking strength tests indicated that the Wurlan finished wool fabrics performed effectively for the length of this study under the conditions used. It is anticipated that such plain and twill weave woolen fabrics would give satisfactory home laundry performance.

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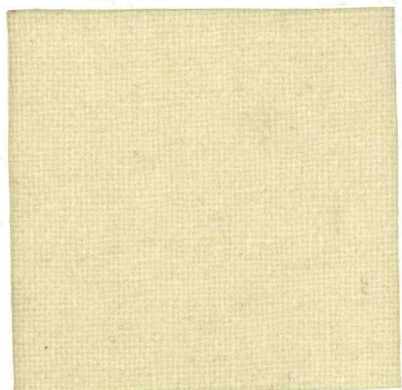
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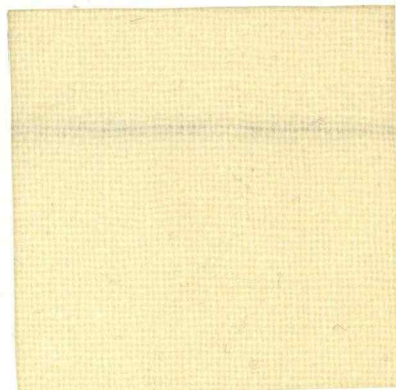
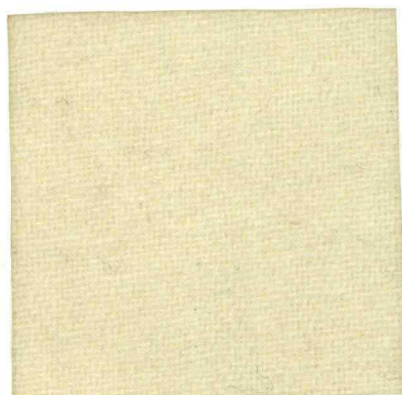
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APPENDIX A

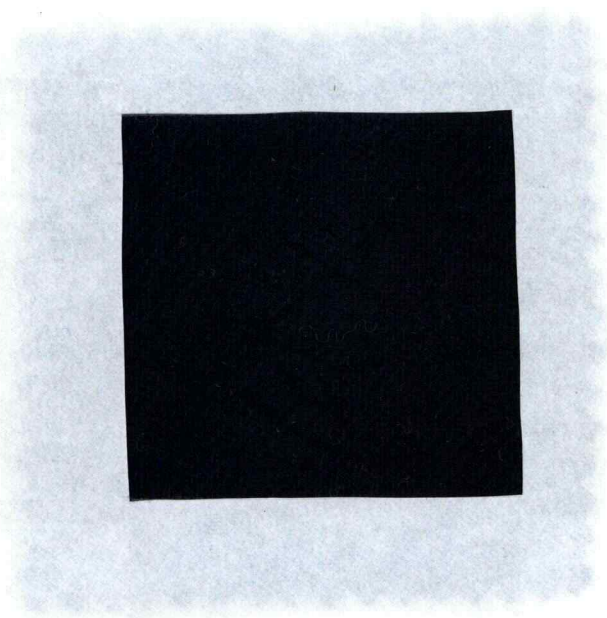
WOOL FABRICS USED IN THIS STUDY



Plain weave, unfinished

Plain weave,
Wurlan finished

Twill weave, unfinished

Twill weave,
Wurlan finished

APPENDICES

APPENDIX B

X-RAY FLUORESCENT SPECTROSCOPY
OPERATING CONDITIONS FOR VARIOUS ELEMENTS

Element	2 θ angle	Target tube	Analyzing crystal	Counter
Bromine	29.98	Tungsten	LiF	Scintillation
Aluminum	112.70	Chromium	EDDT	Flow Counter
Iron	57.52	Chromium	LiF	Scintillation
Silicon	78.20	Chromium	EDDT	Flow Counter
Titanium	86.15	Chromium	LiF	Scintillation

APPENDIX C

LEAST SIGNIFICANT DIFFERENCE IN BREAKING
STRENGTH BETWEEN VARYING CONDITIONS

$$\text{LSD}_{\alpha} = t \left(\frac{\alpha}{2} \right) (v) \sqrt{\frac{2 \text{ MSE}}{r}}$$

$$\text{LSD}_{.05} = t \left(\frac{.05}{2} \right) (80) \sqrt{\frac{2(2.33)}{5}}$$

$$\text{LSD}_{.05} = 2.29 \sqrt{.934}$$

$$\text{LSD}_{.05} = 2.21$$

If there are differences between means of five breaking strength specimens greater than 2.21, they are significant ($\alpha = .05$).

$$\text{LSD}_{.01} = t \left(\frac{.01}{2} \right) (80) \sqrt{\frac{2(2.33)}{5}}$$

$$\text{LSD}_{.01} = 2.90 \sqrt{.934}$$

$$\text{LSD}_{.01} = 2.80$$

Differences of greater than 2.80 between means of five breaking strength specimens are significant ($\alpha = .01$).

APPENDIX D

RESULTS OF SELECTED RAVELLED STRIP BREAKING STRENGTH
TESTS OF WURLAN FINISHED WOOL FABRICS*

Unlaundered	1	2	average
Plain weave, unfinished	16.0	14.5	15.25
Plain weave, Wurlan	23.5	21.5	22.5
Twill weave, unfinished	18.0	19.0	18.5
Twill weave, Wurlan	22.0	20.5	21.25
Laundered with Tide, gentle agitation			
Plain weave, unfinished	~14	14.5	~14.2
Plain weave, Wurlan	20.0	18.0	19.0
Twill weave, unfinished	~14	15.0	~14.5
Twill weave, Wurlan	19.5	20.0	19.75

* lb/sq. in. required to break strips containing 35 warp yarns between jaws measuring 3 in. wide by 2 in. high, under standard conditions ($70 \pm 2^\circ\text{F.}$, $65 \pm 2\%$ relative humidity).

(~ approximately)