PESTICIDE DECONTAMINATION FROM FABRIC BY LAUNDERING FOLLOWING REPEAT CONTAMINATION AND SIMULATED WEATHERING

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CHAPTER I

INTRODUCTION

Historically, chemicals that destroy, prevent, or control pests, have played an important role in pest control As early as 1550 B C preparations controlled pests, and by 900 A D , the Chinese used arsenic to control garden insects (Hays, 1975) Thus, control of pests and diseases that threaten health or food and fiber supply have long been a concern Pests impact on all segments of agriculture causing both a loss of products and an increase in consumer costs (Council for Agricultural Science, 1977) If pesticides were not available, crop losses would exceed the value of all U S agricultural exports (Council for Agricultural Science, 1977) To meet ever increasing demands for food and fibers, agriculturists now rely on a broad range of pesticides including fungicides, insecticides, herbicides, and growth regulators to boost production (Hafen, 1972)

Prior to World War II, primarily natural compounds controlled pests Since then, there has been a significant increase in the development and use of synthetic chemical compounds The early pesticides were commonly composed of chlorinated hydrocarbon chemicals One of these, dichlorodephinyltrichloroethane (DDT) was effective and used extensively in all segments of agriculture (D'Ercole, Arthur, Cain, and Barrentine, 1976) The chlorinated hydrocarbons, when applied to fields, remained toxic for long periods of time Thus, DDT was popular with agriculturists because fewer applications were needed However, the positive attributes of the "Miracle Chemical" soon became clouded with negative ones when residues of DDT were found in human tissues, food, clothing, air, water, and soil (Hafen, 1972) The negative side effects led to a ban of DDT by the Federal, Insecticide, Fungicide, and Rodenticide Act of 1972 (Kilgore and Akesson, 1980)

Two groups of synthetic chemicals, organophosphates and carbamates replaced DDT (Hafen, 1972) Both are frequently more toxic than DDT (Gold, Leavitt, Holcslaw, and Tupy, 1982, Kilgore and Akesson, 1980, Soliman, El-Sebae, and Sorya, 1979) but they break down quickly in the environment and are rapidly metabolized and excreted by mammalion tissue Thus, they are thought to be transient in the environment (Soliman, El-Sebae, and Sorya, 1979)

The use of these new chemicals, particularly the organophosphates, for pest control programs has increased dramatically in both agricultural and domestic settings (Hafen, 1972) One of these new chemical groups, the organophosphates, is frequently criticized because of its possible effect on the human body (Soliman, El-Sebae, and Sorya, 1979) Researchers do not know effects of multiple exposure of pesticides upon the body However, some 20 years ago researchers warned that biological chemicals accumulate over long periods of time and each exposure builds up in the body Subsequent research has linked pesticide exposure to birth defects, cancer, genetic mutations, sterility, liver damage, and disturbances of the nervous system (Alexander, 1960, Green, 1958, Kahn, 1976, Bearn and German, 1961, Egert and Greim, 1976, Infante and Legator, 1979, Depalma, Kwalick, and Zikerberg, 1970, Regan, Setlow, Francis, and Liginski, 1976, Davies, Freed, Enos, Barquet, Morgade, and Danauskas, 1980, Klemmer, Wong, Sato, Reichert, Korsak, and Rashad, 1980) Hence there is mounting concern for the unintentional side effects of pesticides on human health and other forms of life (Boraiko, 1980)

Pesticides may affect the body if inhaled, ingested, or absorbed through the skin Until recently, scientists thought pesticides entered the body primarily by ingestion and inhalation However, Maiback, Feldmann, Milby, and Serat (1981) investigated body absorption of pesticides and reported that the skin can be a major source of human exposure Robbins, Nash, and Comer (1977) also determined that dermal exposure was more significant than respiratory exposure, but the seriousness depended on the toxicity of the pesticide, the rate of absorption of pesticide by body site, the size of the skin area contaminated, and the length of time the pesticide was in contact with the skin

Those handling pesticides may unknowingly encounter health hazards from skin absorption if their clothing becomes contaminated with pesticides Boraiko (1980), Hayes (1975), and Southwick, Mechan, Cannon, and Gortatowski (1974) cite cases of serious illness or deaths that have resulted from individuals wearing pesticide contaminated clothing Once contaminated, clothing may remain a threat to health because ordinary laundry practices may not remove enough pesticide to allow safe wearing (Finley, Metcalfe, McDermott, Graves, Schilling, and Bonner, 1974)

One dramatic example cited by Southwick et al (1974) involved an elderly home gardener who became ill after his clothing became saturated with the pesticide methyl parathion, while spraying fruit trees in his back yard Quick antidotal measures for pesticide poisoning resulted in his recovery A few weeks later, however, the home gardener wore the same set of clothing that had been laundered by regular laundry procedures and became ill again Quick medical attention was not adequate to save his life Cause of death was attributed to pesticide poisoning Subsequent laboratory analysis of the man's clothing revealed high levels of toxic residues of methyl parathion

Because the newer forms of pesticides break down rapidly in the environment, effective pest control programs require frequent applications More frequent handling of pesticides by manufacturers, agricultural workers, gardeners, nurserymen, and domestic users increases the potential for dermal exposure to toxic materials The clothing of these individuals can become contaminated with either concentrated or dilute solutions of pesticides that may range from a fine mist that is undetected to complete saturation due to accidental spills, malfunctioning equipment, or wind drift

Because dermal absorption of pesticides is a possible health hazard, people should emphasize protective clothing as a way to limit dermal exposure A comprehensive study was undertaken to develop and evaluate prototype protective clothing that offered the individual grower protection, thermal comfort, and social acceptability Orlando, Branson, and Henry (1984) provides an overview of this study Part of the study focused on evaluating selected fabrics as barriers to pesticide penetration Tyvek® and Gore-Tex® prevented penetration of the pesticides tested (Orlando, Branson, Ayers, and Leavitt, 1981) Three fabrics and three prototype designs were subsequently evaluated in terms of thermal comfort using human subjects in a controlled environmental laboratory (Branson, DeJonge, and Munson, in press) Physiological and subjective data indicated that the thermal comfort level of subjects wearing Gore-Tex® ensembles was similar to the thermal comfort level of subjects wearing chambray ensembles These results may be due to the impermeable membrane within the structure of the Gore-Tex® that allows the fabric to breathe A subsequent study (Branson, Ayers, and Henry, in press) determined the barrier effectiveness of Gore-Tex® to additional pesticides Based on the findings that Gore-Tex® was both impermeable to the pesticides tested in the penetration studies and thermally comfortable, Gore-Tex® was considered a potential candidate fabric for use in protective garments

Previous studies indicate that the laundry process does not completely remove all pesticide residue from typical work clothing Therefore, there is concern that pesticide residue may build up in fabrics over time Before Gore-Tex® can be recommended for use in protective garments the aspect of 'build-up' must be investigated Additionally, little was known of the effects of different types of surfactants on the removal of pesticide residues from Gore-Tex® Previous pesticide decontamination by laundry studies concentrated on wash and rinse temperatures rather than type of detergents for laundry efficacy There was need for further investigation into the effects of detergents on removal of pesticides from contaminated fabric

Additionally, since pesticides decompose in the natural environment, the possibility exists that exposure to sunlight, heat, and humidity may break down pesticide chemicals in fabric In a field reentry study, Finley, Graves, Summers, Schilling, and Morris (1979) observed that fabric swatches pinned to trouser legs of insect scouts and worn in fields sprayed one day, two days, and four days previously with methyl parathion picked up 90 to 99 percent less residue two and four days after application This suggests that the chemical breaks down quickly in the atmosphere Thus, the question arose as to whether pesticide contamination in fabric could be broken down if exposed to sunlight prior to laundering Thus, this study investigated the phenomena of simulated environmental conditions (weathering) on pesticide breakdown

Purpose

This study consisted of two phases The purpose of Phase I was to determine the percent residue of a commercial-grade pesticide mixture remaining in a test fabric following weathering and detergent treatments The purpose of Phase II was to determine if repeated pesticide contamination and subsequent weathering and detergent treatments influenced pesticide residues remaining in the test fabric

Objectives

The objectives of the study included

1 To determine the effectiveness of detergent treatments on the removal of pesticide from the test fabric

2 To evaluate the effect of simulated weathering on the pesticide removal by laundering

3 To determine whether repeated contamination and subsequent detergent treatments influence pesticide removal from the test fabric

4 To evaluate the effects of repeated contamination, simulated weathering, and detergent treatments on the removal of pesticide from the test fabric

Hypotheses

The null hypotheses for the study were the following

$H_0$1 There was no significant difference ($p < 05$) in the amount of parathion residue remaining in the test fabric after one laundering due to detergent treatments

$H_0$2 There was no significant difference ($p < 05$) in the amount of methyl parathion residue remaining in the test fabric after one laundering due to detergent treatments

$H_0$3 There was no significant difference ($p < 05$) in the amount of parathion residue remaining in the test fabric after one laundering due to simulated weathering

$H_0$4 There was no significant difference ($p < 05$) in the amount of methyl parathion residue remaining in the test fabric after one laundering due to simulated weathering

$H_0$5 There was no significant difference ($p < 05$) in the amount of parathion residue remaining in the test fabric after repeated contaminations and subsequent launderings due to detergent

$H_0$6 There was no significant difference ($p < 05$) in the amount of methyl parathion residue remaining in the test fabric after repeated contaminations and subsequent launderings due to detergent

$H_0$7 There was no significant difference ($p < 05$) in the amount of parathion residue remaining in the test fabric after repeated contaminations and subsequent launderings due to simulated weathering

$H_0$8 There was no significant difference ($p < 05$) in the amount of methyl parathion residue remaining in the test fabric after repeated contaminations and subsequent launderings due to simulated weathering

Limitations

1 One pesticide, one formulation, and one concentration was selected for this study

2 One laundry water temperature was selected

3 One test fabric was evaluated

Assumptions

1 The pesticide used in the study was representative of one frequently used in Oklahoma agriculture

2 The laboratory equipment and laundry procedures simulated one home laundering

Definitions

Anionic Surfactant A detergent carrying a negative charge They ionize in solution and are generally high sudsing (Soaps and Detergents, 1981)

Builder A substance that aids cleaning efficiency Examples include fluorescent whiteners, antideposition agents, corrosion inhibitors, suds control agents, and others (Soaps and Detergents, 1981)

Cationic Surfactant A detergent carrying a positive charge that ionizes in solution They are commonly used in fabric softeners (Soaps and Detergents, 1981)

Degradation The breakdown of a complex chemical by the action of microbes, water, air, sunlight, or other agents (Bohmont, 1981)

Emulsion A mixture in which one liquid is suspended as minute globules in another liquid (Bohmont, 1981)

Emulsifiable Concentrate The mixture produced by dissolving a toxicant (pesticide) and an emulsifying agent in an organic solvent (Farm Chemical Handbook, 1979)

Encapsulated Pesticide "Pesticides enclosed in tiny capsules of such material so as to control release of chemical and extend the period of diffusion, thus, providing increased safety to applicators and the environment" (Farm Chemical Handbook, 1979, p 164)

Gore-Tex® A three layer fabric with an outer layer of rip-stop nylon, an inner layer of nylon tricot, and a microporous film of polytetrafluorecethylene laminated between two other fabric layers

Laundry Detergent A product containing a surfactant and other ingredients, formulated to clean and care for the fabrics (A Handbook of Industry Terms, 1981)

Methyl Parathion An insecticide The chemical name is 0,0-Dimethyl-0-p-nitrophenyl phosphorothioate The toxicity acute oral LD50, acute dermal 50 It is extremely toxic to warm blooded animals through inhalation and skin absorption

Nonionic Surfactant A detergent that does not ionize in solution and has no electrical charge Nonionic detergents are resistant to water hardness and clean well on most oily soils (Soaps and Detergents, 1981)

Parathion An insecticide The chemical name is 0,0-Diethyl-0-p-nitrophenal phosphorothioate Toxicity acute oral LD50, acute dermal 50 It is extremely toxic to warm blooded animals through inhalation and skin absorption

Pesticide

> Any substance or mixture of substances intended for preventing, destroying, repelling, or mitigating any insects,

> rodents, nematodes, fungi, or weeds, or any other forms of life declared to be pests, and any substance or mixture of substances intended for use as a plant regulator, defoilant, or desiccant" (Bohmont, 1981, p 41)

<u>Pesticide Residue</u> The amount of chemical which remains on a product (Bohmont, 1981)

<u>Phosphates</u> A complex group of sequestering agents used in detergent formulations because of their superiority in water softening, sequestering, and other builder functions (<u>Soaps and Detergents</u>, 1981)

<u>Surfactant</u> (surface active agent) Surfactants are wetting agents that lower the water's surface tension allowing water to penetrate fabrics and remove water soluble soils (<u>Soaps and Detergents</u>, 1981)

<u>Weathering</u> Exposure to atmospheric elements, such as temperature, moisture, air, cloudiness, sunlight, and wind

<u>Wettable Powder</u> A solid (powder) formulation that forms a suspension used for spraying when added to water (Bohmont, 1981)

CHAPTER II

REVIEW OF LITERATURE

The literature review focused on factors pertinent to removal of pesticide by laundering A discussion of factors important to oily soil removal from textiles was included because such factors were expected to influence removal or retention of parathion and methyl parathion from Gore-Tex® fabric No research investigating repeat pesticide contamination of fabric samples was found

Decontamination by Laundry

Soil removal from fabric has been most commonly attempted through the laundry process with an aqueous medium and detergent Kissa (1981) defined the laundry process in three consecutive steps including induction period, mechanical dislodgement period, and soil removal period During the induction period, water and detergent surfactants (Surface Active Agents) or wetting agents break up surface tension of water and allow the laundry liquid to diffuse into the soiled fabric This period is facilitated by agitation, type of detergent, type of surfactant, and type of soil Because only liquid soil can separate spontaneously from fabric in an aqueous medium, solid soils require mechanical action to dislodge and transport soil into the laundry liquor In the soil removal period surfactants inhibit or reduce soil adhesion to fabrics and suspend soil where it can be carried away in the laundry water

Wash Water Temperatures

Research investigating decontamination of pesticides from fabrics has indicated that wash water temperatures influence removal rates of pesticide Lillie, Hamilton, Livingston, and Porter (1980) assessed the effect of 30°C, 40°C, and 60°C laundry water temperatures for removal of concentrated solutions of malathion, bromacil, diazinon, chlordane, and propoxur, and dilute solutions of diazinon, chlordane, and propoxur from 100 percent cotton coveralls Samples were laundered in an automatic washer with one 14-minute wash cycle and two 5-minute rinse cycles The laundry liquor with a pH of 11 consisted of tap water and one-half cup nonphosphate detergent containing carbonate, silicon, sulfonate, sodium sulfate, and alkyl aryl sulfonate

These researchers found that 80 percent of all the concentrated pesticide solutions tested were consistently removed by laundering regardless of wash water temperatures However, hot (60°C) water was most effective and removed 96 percent or more of all the tested pesticides Chlordane behaved differently than other pesticides in that cold water removed 99 9 percent of the concentrated solution

Kim, Stone, and Sizer (1982) evaluated 60°C (hot), 49°C (warm), and 40°C (cold) wash temperatures with and without detergents to remove concentrated fonofos and alachlor from lightweight and heavyweight 100 percent cotton denim fabrics Samples were laundered for three minutes, then rinsed three and five minutes in a launder-ometer, with distilled water, and a commercial phosphate detergent The researchers found that no distinct pattern of pesticide removal could be attributed to wash water temperature Their findings conflicted with the findings of

Lillie et al (1980) that hot (60°C) wash water plus detergent removed 96 percent of all the pesticides tested However, different pesticides were used

Easter (1982) assessed three wash and rinse water temperature combinations for removal of captan and Guthion® from the fabrics Gore-Tex®, Tyvek®, and two 100 percent cotton fabrics Selected wash and rinse water temperatures were 38°C and 28°C, 49°C and 38°C, and 60°C and 49°C Samples were laundered 12 minutes in a launder-ometer with a laundry liquor made up of a non-phosphate laundry detergent containing both anionic and nonionic surfactants, water with 150 parts per million (ppm) hardness and pH level of 9 20± 0 10 The rinse water had the same grains of hardness as the wash water but a pH level of 5 2± 0 10 Captan residue retained in the test fabrics ranged from 27 26 percent in 38°C wash water to 0 05 percent in 60°C wash water Wash water temperatures, likewise, affected removal of Guthion® from test fabrics Guthion® residue ranged from 13 56 percent in 38°C wash water to 0 06 percent in 60°C wash water The results indicated that an increase in wash water temperature resulted in greater removal of both captan and Guthion® from all test fabrics Easter's findings support those of Lillie et al (1980) that increased temperature of wash water removed greater amounts of pesticide

Breen, Durnam, and Obendorf (1984) evaluated wash water temperatures of 4°C, 27°C, 38°C, and 49°C with three detergents for the removal of oily soil from 50/50 percent polyester/cotton fabric samples contaminated and laundered five times before analysis The oily soil was composed of 50 percent tristearin, 40 percent oleic acid and 10 percent octadecane diluted with toluene The detergents used were a heavy duty powdered detergent containing an anionic surfactant with sodium carbonate and zeolite builders, a heavy-duty powdered detergent containing an anionic surfactant with sodium carbonate builders formulated for all wash water temperatures, and an unbuilt heavy-duty liquid containing both anionic and nonionic surfactants. Breen, Durnam, and Obendorf (1984) found that the oily soil retained in the test fabric ranged from 2.21 percent at 4°C to 0.96 percent at 49°C.

Prato and Morris (1984) evaluated the effect of combined variables of detergent concentration, agitation time, and wash water temperatures for the removal of oily soil on 100 percent acrylic, cotton, nylon, and polyester fabrics. The fabric samples were washed in water with 120 ppm hardness and a detergent containing an anionic surfactant with phosphate. Samples were soiled with oily soil composed of tristearin (23%), triolein (23%), stearic acid (15%), oleic acid (15%), squalane (8%), hexadecanal (8%), and cholesterol (8%) and laundered twice before soil retention was measured. The amount of soil that remained on the fabric after laundering was determined by measuring fabric whiteness. Agitation times were 5, 7, 10, 13, and 15 minutes, wash water temperatures were 21°C, 29°C, 41°C, 52°C, and 60°C, and detergent concentrations were 0.05, 0.09, 0.15, 0.21, and 0.25 percent of the wash water volume.

The researchers found agitation time to be less significant than detergent concentration or laundry temperatures for removing the oily soil. Detergent concentration of 0.20 percent, and wash water temperature of 21°C removed oily soil from the nylon and acrylic fabrics while 0.25 percent detergent and a wash water temperature of 41°C was the optimum combination required to remove oily soil from the polyester and cotton. They credited the alkaline wash liquid formed by the built detergent for better oily soil removal because the alkaline solution allowed the soil to dissolve in water. However, only one detergent was used in the study.

Wash water temperature effect on soil retention varied with fiber content. Wash water temperature of 52°C resulted in a whiteness index of 97.1 for 100 percent cotton, while 41°C resulted in the highest (59.3) whiteness index for acrylic. Wash water temperatures of 29°C resulted in a whiteness index of 69.6 for nylon and 61.8 for polyester. The authors credited the 97.1 whiteness index for cotton to the affinity of the detergent's fluorescent brighteners for cotton fiber rather than to better soil removal. Both Breen, Durnam, and Obendorf (1984) and Prato and Morris (1984) found that hot water was most effective in removing soil from fabrics containing cotton. However, brighteners found in detergents may have influenced results of Breen, Durnam, and Obendorf (1984) as well as Prato and Morris (1984). Thus, pesticide removal studies by Lillie et al. (1980) and Easter (1982) agreed with the findings of the oily soil removal studies by Breen, Durnam, and Obendorf (1984) which found that higher wash water temperatures removed oily soil best. Fiber content and concentration of detergent were important factors in soil removal. Detergent concentration was not varied in pesticide studies.

Laundry Detergents

Southwick, Mecham, Cannon, and Gortatowski (1974) evaluated cationic, and anionic surfactants and bleach for removing both concentrated and field strength solutions of DDT, aldrin, dieldrin, chlordane, diazinon, duraban, and parathion from work coveralls. The fiber content was not reported These researchers washed some samples at a laundromat but because machine performance varied extensively, glass flasks and a shaker were used to launder most of the samples They found that the class of detergent influenced residue removal Anionic detergents, phosphate content unknown, removed less than 50 percent of the methyl parathion but bleach removed 95 percent Their report did not clearly specify whether bleach was used with the detergent Cationic detergent removed only 15 percent of the methyl parathion The authors concluded that methyl parathion contaminated fabric may not be safe for wearing after only one laundering

Easley, Laughlin, Gold, and Schmidt (1982) evaluated four detergents and three combinations of wash and rinse water temperatures for the removal of emulsifiable concentrate methyl parathion from two denim fabrics of 100 percent cotton and 50/50 percent cotton/polyester Three detergents selected represented common categories of commercial preparations They were phosphate, carbonate, and heavy-duty liquid The type of surfactant was not reported, and it was not clear whether the carbonate and heavy-duty liquid were phosphate free The fourth was an AATCC Standard Detergent 124 with 12 percent phosphate All detergents tested removed similar amounts of methyl parathion Because there was no clear indication that any one type of detergent was significantly better than another for removal of methyl parathion, the authors concluded that factors other than detergent were important to pesticide removal Though not statistically significant, there was a trend for more effective removal of methyl parathion at wash water temperatures of 49°C and 60°C with the heavy-duty liquid detergent The authors credited the heavy-duty liquid's oil removing ability for its efficiency in removal of the oil based emulsifiable concentrate although they did not specify the detergent surfactant nor whether the detergent contained phosphates

Easley, Laughlin, Gold, and Tupy (1983) assessed four laundry treatments in 60°C wash water for removal of three formulations of methyl parathion from denim fabrics of 100 percent cotton and 50/50 percent cotton and polyester The laundry treatments used were a two-minute pre-rinse followed by AATCC Standard Detergent, detergent with 12 percent phosphate, a phosphate detergent wash, an ammonia (NH3) additive plus phosphate detergent, and bleach (NaOCL) plus a phosphate detergent The most effective laundry treatment for removing methyl parathion, regardless of fiber content, was the pre-rinse plus the detergent Methyl parathion removal ranged from 87 8 to 99 9 percent The least effective laundry treatment was the ammonia and detergent combination which removed a range of 80 6 to 96 3 percent Researchers observed that factors other than detergents, such as pesticide formulation, impacted on pesticide removal Detergent and ammonia removed less of the emulsifiable concentrate than other treatments Fifty-nine to 68 4 percent of the emulsifiable concentrate was removed as compared to 75 5 to 81 5 percent of the wettable powder and encapsulated crystal formulations

Fabric Properties

Fiber Content

Removal of pesticides from fabric through the laundry process is influenced by fiber content, fiber length, and cross-sectional shape, finishes, and fabric weight Finley and Rogillio (1969) evaluated laundering as a means of removing methyl parathion and DDT from five shirt-weight fabrics of twill weave Four of the test fabrics were polyester and cotton blends 35 percent cotton/65 percent polyester, 50 percent cotton/50 percent polyester, 65 percent cotton/35 percent polyester with a durable press finish, and 65 percent cotton/35 percent polyester with both a durable press and a soil release finish The fifth fabric was 100 percent mercerized and sanforized cotton Swatches of the five test fabrics were contaminated with a combined standard solution of methyl parathion and DDT Contaminated swatches were laundered and dried in an automatic washer and dryer according to AATCC Method 124-1967 Neither the detergent surfactant nor phosphate content were reported After contaminated samples were laundered once, the methyl parathion retained in the blended fabrics ranged from 0 8 percent in the 50/50 percent cotton/polyester fabric to 0 6 percent in the 65/35 percent cotton/polyester fabric Methyl parathion retained in the 100 percent cotton fabric was 6 3 percent The researchers found that the greater the cotton content, the higher the amount of methyl parathion retained after one laundering Laundry was less effective for removal of DDT from all fabrics In the blended fabrics, DDT residue ranged from 12 5 percent in 50/50 polyester/cotton fabric to 21 2 percent in the 65/35 cotton polyester fabric In the 100 percent cotton, 20 2 percent remained Again the higher the cotton content the greater the DDT retained after one laundering and drying They did not report the wash water temperature but credited the more effective removal of methyl parathion from test fabrics to the laundry liquor The detergent used produced an alkaline wash liquor with a pH of 10 and methyl parathion is hydrolized to p-nitrophenal in an alkaline medium

The results of the Easley et al (1982) study conflict with those of Finley and Rogillio (1969) in that Easley et al (1982) concluded that there was no significant difference between 100 percent cotton and 50/50 percent cotton/polyester in retention of methyl parathion after one laundering Easter (1982) found that captan in wettable powder form was more difficult to remove from the natural fiber cotton than from the synthetic fabrics The range of pesticide retained in the 100 percent cotton denim and chambray fabric was 27 26 to 0 05 percent as compared to 2 24 to 0 00 percent in the synthetic Gore-Tex® and Tyvek®

In the same study Easter (1982) found the pesticide Guthion® more difficult to remove from the synthetic fabric Gore-Tex® than from Tyvek® or the cotton fabrics The residue retained in the Gore-Tex® fabric was 13 56 to 2 10 percent, and 5 10 to 0 19 percent in the Tyvek® fabric The 100 percent cotton denim and chambray retained residues ranging from 5 74 percent to 0 06 percent Easter concluded that pesticide removal was influenced by the combined variables of fiber, fabric composition, laundry treatments, and pesticide properties

Fiber Length and Cross-Sectional Shape

In a laundry study, Brown, Thompson, and Stewart (1968) found that fiber content as well as fiber length influenced oily soil removal They evaluated 100 percent polyester filament, 100 percent nylon filament, 100 percent staple polyester and 100 percent cotton fabrics for the removal of grease, heavy lubricating oil, light cooking oil, olive oil, vegetable cooking oil, sebum-type oil, and linseed oil They found that when polyester staple and polyester filament fabrics were oil soiled, the staple polyester fabric was more difficult to clean than the polyester filament fabric After five launderings 85 percent of the oil was removed from the staple polyester fabric, but 96 1 percent was removed from the filament polyester fabric They found that the optimum wash water temperature for removal of oily soil was dependent upon the fiber length The 100 percent polyester filament fabric was washed clean in 48°C wash water, while the staple polyester fabric cleaned better at 85°C rather than at 48°C The authors concluded that the higher wash water temperatures were more effective for removing oily soils from polyester staple fabric The cross-sectional shape of the polyester was not reported In a related study, Obendorf, Namaste, and Durnam (1983) found that the increased surface area of trilobal polyester filaments made soil removal difficult because the interfiber capillary systems formed closed spaces that trapped soils

Obendorf, Namaste, and Durnam (1983) investigated residual oily soil distribution on fabrics in relation to fiber content and cross-sectional shape Fibers investigated were acrylic, polyester (round), polyester (trilobal), nylon, cotton with durable press finish, polyester/cotton with no finish, and polyester/cotton with durable press finish All samples were soiled with triolein in toluene, aged at 38°C for 16 hours, and laundered with a heavy-duty unbuilt detergent The synthetic fibers retained different quantities of oily soil after laundering, but the researchers found no difference in the location of the residual soil The nylon and acrylic fibers retained beads of oil on the fiber surface while polyesters (round and trilobal) retained an even coat of oil on the fiber surface No oily soil was found in the interior of any of the synthetic fibers In the cotton fibers, oily soil was found within the lumen of the fiber The distribution of oily soil residues on the polyester/cotton blends without a resin finish and on the blend with the resin finish was the same The blend with the resin finish retained less oily soil residue after laundering than did the blend with no resin finish The resin finish prevented the oily soil from adhering to the fiber surface The trilobal polyester fabric retained more oily soil than did the polyester fiber with the round shape The authors credited the difference in oily soil retention to the cross-sectional shape of the fiber The trilobal fiber had an increased surface area and close spaces within the yarn structure where water and detergent were inhibited from reaching all of the fiber surfaces

Fabric Finish

Fabric finishes affect adherence of pesticide residue to fabric surfaces Solberg and Obendorf (1985) used an electron microscope to determine the distribution of the pesticide malathion on and within 50/50 cotton/polyester fabrics with and without a durable press finish The fabrics were laundered once with a detergent containing an anionic surfactant Cross-sectional fiber analysis of both the unfinished and durable press finished fabrics revealed an accumulation of malathion in the lumen of the cotton fibers However, the location of a cotton fiber within a yarn structure influenced the amount of malathion found in or on the surface of a cotton fiber Fibers on the surface of the yarn had greater amounts of malathion both on the surface and in the center of the yarn Polyester fibers retained a thin film of malathion on the outer fiber surface but none within the interior of the fiber regardless of the fiber's position within the yarn Thus, all polyester fibers within a yarn retained similar amounts of malathion The durable press finish did not alter the distribution of the malathion on the surface of the polyester fiber, the surface of the cotton, or within the lumen of the cotton However, in the unfinished fabric, more malathion was found on the surface of both the cotton and the polyester fibers Thus, the authors concluded that fiber content was an important factor in pesticide decontamination The cotton fibers absorbed the pesticide into the center of the fiber, while the polyester fibers allowed the malathion to penetrate through the fabric

Obendorf and Solberg (1985) investigated the distribution of malathion and methyl parathion on fabrics unlaundered and laundered once The fabrics consisted of both unfinished and durable press finished 50/50 polyester/cotton The detergent used contained an anionic surfactant with carbonate and zeolite builders In the unlaundered samples, they found that malathion and methyl parathion residues were present on the fiber surfaces, and the pesticide residues were distributed evenly throughout the polyester yarns Neither pesticide was found within the interior of the polyester fiber Cotton fibers located near the surface of a yarn retained greater amounts of malathion and methyl parathion residue than did fibers located in the center of a yarn These findings agreed with a previous pesticide residue microscopical distribution study by Solberg and Obendorf (1985) who found fiber content, as well as fiber location within a yarn, important to pesticide removal by laundering

In the polyester fabric samples, laundered one time, a microscopical analysis indicated remaining pesticide residue was uniformly distributed on the fiber surface throughout the yarn in both the unfinished and finished fabrics In the cotton fabric, the laundry treatment removed the pesticides from the fiber surface more effectively in fibers located near or on a yarn's surface Laundering did not remove the malathion or methyl parathion from the lumen of the cotton fiber The authors indicated that removal of the pesticide residues from the lumen of cotton fibers may not be possible with an aqueous process

Keaschall, Laughlin, and Gold (1984) assessed 50/50 percent polyester/cotton test fabrics with three finishes for removal of emulsifiable concentrate formulations of organophosphate, carbamates and organochlorine classes of pesticides One fabric did not have a finish, a second had a renewable consumer applied fluorocarbon finish, and the third had a commercial soil-repellent fluorocarbon finish After one laundering, the mean residues retained across all pesticides tested ranged from 0 63 µg/cm in the unfinished fabric to 0 06 µg/cm in the renewable fluorocarbon finished fabric and 0 16 µg/cm in the fabric with the commercial soil-repellent finish The authors found a significant interaction between fabric finish and pesticide class An analysis of the main effect pesticide class indicated that organochlorine pesticides were the most difficult to remove by laundry, followed by organophosphates and carbamates The mean residue retained by pesticide class was from 5 56 percent for organochlorines to 3 49 percent for organophosphates and 0 10 percent for carbamates Because of the interaction of finish and pesticide the authors could make no clear conclusion regarding the impact of fabric finishes on pesticide retention

Fabric Weight and Construction

Kim, Stone, and Sizer (1982) evaluated shirt-weight and pants-weight 100 percent cotton denim for the removal of concentrated emulsifiable solutions of the pesticides fonofos and alachlor. The researchers found that light-weight fabrics retained 41.9 percent of the fonofos and 2.2 percent of the alachlor while the heavy-weight fabrics retained 80.7 percent of the fonofos and 46.7 percent of the alachlor. The authors suggested that pesticide removal may be more difficult with the heavier fabrics because the thickness and weight may allow deeper penetration of the pesticide into the fibers and fabric structure through the wicking process. These findings are supported by the Obendorf, Namaste, and Durham (1983) study that found that the intercapillary system formed close spaces that could trap soil and prevent removal by laundering in an aqueous medium.

Bowers and Chantrey (1969) evaluated four 100 percent cotton fabrics of different weights and fabric constructions for the removal of oily soil with a nonionic detergent. The fabrics used were oxford (4.0 ounces per square yard), broadcloth (3.1 ounces per square yard), poplin (6.8 ounces per square yard), and twill (7.1 ounces per square yard). They found that fabric weight and fabric construction had an effect of soil removal. The loosely woven oxford cloth (weight 4.0 ounce per square yard) containing low twist yarns retained 16 percent of the original soil contamination while broadcloth, a tightly woven cloth (weight 3.1 ounce per square yard) made of high twist yarns retained 23.2 percent of the original oily soil. The poplin and the twill, both tightly woven fabrics of high twist yarns, retained 27.7 percent and 32.1 percent respectively.

Pesticide

Solubility

A search of the literature revealed that pesticide class, solubility, and formulation may have a significant influence upon pesticide removal from fabric Easley et al (1983) evaluated 80/20 percent cotton/polyester blend denim fabric and 50/50 polyester/cotton double knit t-shirt fabric for removal of 2,4-Dichlorophenoxyacetone acid ester and 2,4-Dichlorophenoxyacetic amine herbicide compounds by laundry The 80/20 percent cotton/polyester fabric was pesticide contaminated prior to simultaneous laundry with 50/50 percent cotton t-shirt fabric

The researchers detected differences in removal rates between the two pesticide compounds Results show that 2,4-D ester, a compound insoluble in water, was difficult to remove by laundry methods The mean percents retained in the denim fabric following one laundering ranged from 71 14 percent to 55 01 percent Residues of 2,4-D amine, a compound readily soluble in water, ranged from 0 83 to 0 35 percent in the contaminated denim fabric The authors attributed the low level of pesticide residue to 2,4-D amine's water solubility They concluded that the water soluble 2,4-D amine compound was more effectively removed by laundering than the insoluble mixture The findings of Easley et al (1983), Breen, Durnam, and Obendorf (1984), and Prato and Morris (1984) are in agreement with Easter's (1982) results suggesting that pesticides formulated with an oil base were difficult to remove from synthetic fabrics The findings of Easley et al (1983), Easter (1983), and Kim, Stone, and Sizer (1982) are in agreement with Lillie's et al (1980) study that found pesticide removal may be dependent upon the specific pesticide These studies investigated several different pesticides but with the exception of the 2,4-D herbicides the solubility was not reported

Formulation

Pesticide formulation affects the removal of contamination from fabric Laughlin, Easley, Gold, and Tupy (1981) evaluated three formulations of methyl parathion emulsifiable concentrate, encapsulated crystals, and wettable powder in a laundry study They found that wettable powder formulations were more easily removed by laundry than emulsifiable concentrates or encapsulated crystals The authors suggested that the particulate nature of the wettable powder might contribute to easy removal Easley et al (1981) found emulsifiable concentrate formulations more difficult to remove than either encapsulated crystals or wettable powder formulations One laundering removed 93 to 99 percent of the encapsulated crystal or wettable powder formulations but only 80 to 88 percent of the emulsifiable concentrate formulation Thus, Laughlin et al (1981) and Easley et al (1981) agreed that wettable powder formulations were easier to remove from test fabric than the emulsifiable concentrate formulation

Degradation

Pesticides are known to degrade in the natural environment (Davis, Staiff, Butler, and Armstrong, 1977, Kawar, Gunther, and Iwata, 1978, and Sharom and Miles, 1981) Chemical degradation may be an important factor in pesticide removal from fabrics Thus, the time lapse and the storage conditions between contamination and laundering may influence pesticide residues

Easter (1982) found that pesticide removal increased if captan contaminated test fabric was stored in covered canisters and held at temperatures ranging from 25° to 30°C for seven days prior to laundering She concluded that the pesticide underwent some chemical or physical change during storage Kim, Stone and Sizer (1982) however, found that laundering swatches immediately following contamination removed significantly greater residues than when washing was delayed 24 hours The results of these two studies appeared to conflict but the latter researchers did not measure degradation of the chemicals

Limited research was found regarding the degradation of pesticides in fabric Easter (1982) evaluated the effect of weathering and laundering on the breakdown of the wettable powdered formulation of captan in two 100 percent cotton and two synthetic fabrics She found that fabric samples exposed to weathering and laundering retained captan residues ranging from 91 72 percent in the cotton denim to 99 00 percent in the Gore-Tex® fabric The unweathered and laundered fabric samples retained captan residues ranging from 97 76 percent in 100 percent cotton denim to 99 00 percent in the Gore-Tex® She concluded that weathering did not breakdown the captan in fabric samples

The following studies have been included in this research to support the use of weathering as a possible means of breaking down pesticide contamination in fabrics before being laundered Sharom and Miles (1981) investigated the breakdown of parathion in an aqueous medium Parathion was added to bottles of 'natural water' collected from a drainage canal Bottles of parathion contaminated water were capped, shaken one minute, and stored at 21±1°C (room temperature) At selected time intervals aliquots were removed from bottles and analyzed They found that 90 percent of the pesticide remained after one week, but disappeared completely after two weeks The authors credited the microorganisms found in the 'natural water' for the degradation of parathion and they felt the low level of degradation of pesticide (10%) the first week was due to the microorganisms not being adapted to react with the parathion to promote the break down of the pesticide Once the microorganisms made the adaptation, the pesticide degraded rapidly

Davis et al (1977) determined the persistence of methyl parathion spilled on cement blocks exposed to indoor and outdoor conditions where no attempt was made to clean up the spill Natural outdoor conditions of the Pacific Northwest portion of the United States were used The average rainfall was 25 centimeters per year and the average number of days with sunshine was 275 The temperature for 14 days in summer averaged 32°C The blocks were covered with snow for two months in the winter Swab samples, collected by gauze pads soaked in alcohol, were taken periodically for one year and analyzed They found that the pesticide in swab samples taken from the blocks exposed to sunlight persisted for three months, while indoor samples retained methyl parathion for four and one-half months Thus, the indoor samples protected from sunlight and moisture retained the methyl parathion one and one-half months longer than the outdoor exposed samples

A second part of the study followed the same methods as the first part except clean-up of the spill was attempted Dirt was piled on the blocks and left for 15 minutes to soak up the pesticide Dirt was removed and the block was flushed with water while being scrubbed with a stiff brush for 30 seconds The blocks were allowed to dry for 24 hours before any swab samples were taken In the outdoor-exposed methyl parathion contaminated blocks with a smooth surface, the methyl parathion decayed from 70 $\mu g/cm^2$ after one day to 2 $\mu g/cm^2$ at two and one-half months Then the weather turned cold and wet, and the level of methyl parathion rose again Surprisingly, at four and one-half months after initial contamination, the measurement level of methyl parathion rose to 5 $\mu g/cm^2$ The pesticide decayed completely six and one-half months after initial contamination In the same experiment but with the pesticide ethyl parathion, noticeable differences in persistence were noted The level of contamination after one day was 70 $\mu g/cm^2$ and after two and one-half months the level dropped to 35 $\mu g/cm^2$ When the weather turned cold and damp the measurable level of ethyl parathion rose to 50 $\mu g/cm^2$ Fifteen months were required for the ethyl parathion to completely decay Thus, the length of time required for pesticides to decay varies with the pesticide and the environmental conditions, such as moisture, heat, and sunlight The persistence of the indoor-exposed ethyl parathion was not measured

Kawar, Gunther, and Iwata (1978) determined the persistence of parathion in wine Reconstituted grape concentrate and 160 milligrams of parathion (25 ppm) were placed in heat sterilized jars and shaken for 36 hours The jar lids allowed carbon dioxide to escape The mixture fermented for 12 days at 20°C before the supernatant wine was siphoned off After two additional weeks the supernatant was again siphoned off and stored for one month The completed wine was filtered through paper, bottled, corked, and stored samples were drawn at intervals of 12, 26, and 56 days during processing for measurement of parathion The level of parathion decreased from 25 5 ppm on day zero to 10 3 ppm at 12 days, 9 0 at 26 days, and 8 8 ppm at 56 days The dregs (lees) at the bottom of the wine container contained much higher levels of parathion than the supernatant After 12 days the lees contained 156 ppm but dropped to 23 ppm at day 26 The authors credited the high level of pesticide found in the lees to absorption of the pesticide by the lees They attributed much of the decrease of parathion in the wine to the fact that the lees laden with pesticide settled to the bottom of the container and were discarded after siphoning off the supernatant The temperature and storage conditions of the wine during processing were not reported

In field re-entry studies, Wolfe, Armstrong, Staiff, Comer, and Durham (1975) measured potential dermal exposure levels of parathion to hand apple thinners Parathion was applied to apple orchards in Washington during June They found parathion residues of 4 0 $\mu g/cm^2$ after one hour, 2 4 $\mu g/cm^2$ after 24 hours, 1 7 $\mu g/cm^2$ after 48 hours, 0 8 $\mu g/cm^2$ after 72 hours, and 0 6 $\mu g/cm^2$ after 96 hours

Draper and Street (1981) measured methyl parathion residues on alfalfa leaves 4, 28, and 72 hours after spraying They found that methyl parathion residues of 2 8 micrograms/kilogram four hours after spraying, 0 71 mg/kg after 28 hours, and 0 32 mg/kg after 72 hours

The results of studies by (Draper and Street, 1981, Davies, Freed, Enos, Barquet, Morgade, and Danauskas, 1980, Kawar, Gunther, and Iwata, 1978, Sharom and Miles, 1981, Wolfe et al , 1975) indicated that the time interval required for parathion and methyl parathion to break down in the presence of a variety of environmental conditions varied from a few hours to 15 months

Summary

A review of the literature suggested that many variables influence the amount of chemical residue remaining in fabrics after laundering Those variables included laundry factors of wash water temperature, detergent type, fabric properties, and pesticide properties

Wash water temperatures were significant for removal of pesticides and oily soil from fabrics Wash water temperatures of at least 60°C were best for removing most pesticides from cotton and cotton/polyester blends However, the fiber length and cross-sectional shape of a fiber may have significant influence in pesticide and oily soil removal Staple fibers and trilobal fibers were more difficult to clean than filament and round fibers There was an indication that higher wash water temperatures (up to 85°C) were necessary for removal of soil from staple polyester fibers and trilobal fibers

A variety of laundry products have been used in pesticide removal studies Anionic, nonionic, and a combination of the two surfactants have been used in the powdered and liquid form both with and without phosphates or other builders The findings do not clearly indicate which if any of the detergent types best removed either pesticide or oily soil

Cotton and cotton/polyester blended fabrics were used as test fabrics for the majority of the pesticide removal studies Only one pesticide decontamination study investigated nylon fabric The synthetic fibers were generally easier to decontaminate of pesticide than cotton fabrics However, factors other than fiber content apparently had significant influence on removal or retention of pesticide residue Those factors included fiber length and cross-sectional shape, fiber weight and construction, and properties of individual pesticides

Pesticide properties influenced pesticide removal regardless of fabric properties or laundry variables Though testing of water soluble and water insoluble pesticides were limited, the 2,4-D herbicides in the water soluble form were readily removed by laundry, while the water insoluble forms were difficult or impossible to remove A wide variety of individual pesticides were tested, yet removal appeared to be tied to properties of the specific pesticides Pesticide formulation was also an important factor in removal Emulsifiable concentrate formulation was generally easier to remove than the encapsulated crystal formulation or wettable powder formulations from synthetic fabrics but was difficult to remove from cotton fabrics Apparently, the staple cotton fibers and the crenulations of the cotton fiber trapped the particulate, wettable powder formulation, making it difficult to remove Synthetic staple fibers, also, appeared to trap the wettable powder in the inter-capillary system making removal difficult

CHAPTER III

METHODOLOGY

This chapter presents a description of the equipment and procedures used for the research. Included are the experimental design, fabric properties and preparation, pesticide, contamination of samples, weathering, laundry detergent treatments, extraction, pesticide residue analysis, gas chromagraph, and the statistical analysis.

Experimental Design

The independent variables of this split plot design included two weathering levels, exposure and nonexposure to simulated environmental conditions, and three detergent levels, anionic, nonionic and combined anionic/nonionic surfactants. A 2x3 factorial design was used to test the effect of weathering and detergent on the percent pesticide residue (dependent variable) retained by the test fabric. A 2x3 factorial design was used to test the effect of weathering and detergent on pesticide residue retained in the test fabric after five repeat pesticide contaminations and subsequent treatments.

In the split plot design three weathered samples and three unweathered samples were contaminated at different times but all were laundered at the same time. Each set of three contaminated samples formed a split plot and the pesticide was randomly applied within each plot The data were blocked by replication and each replication was completed on successive days

Test Fabric

The test fabric was a three layer fabric with an outer layer of rip-stop nylon, an inner layer of nylon tricot, and a microporous film of polytetrafluorecethylene (PTFE) laminated to the two other fabric layers

Fabric Thickness

ASTM D1777-64 was followed to determine the thickness of the test fabric Ten randomly cut conditioned samples measuring five centimeters by five centimeters were measured with a thickness gauge The calculated mean thickness was 0 01 millimeter

Fabric Weight

ASTM D1910-75 was followed to determine the average weight of the test fabric specimens Five randomly cut conditioned samples measuring five centimeters by five centimeters were weighed or a Torsion Balance Model #DRX2 The calculated mean of 3 76 grams was used to determine the weight per square meter which was 1457 1713 grams

Fabric Preparation

Prior to the study, the test fabric was laundered to remove manufacturer's sizing Because three different detergents were used in the study, the fabric was divided into three parts and each part laundered one complete cycle in a Maytag Automatic washer Model #LA312 in one of the three detergents Fabric was dried 15 minutes in a Maytag Automatic dryer Model #LDE712 set for permanent press

The test fabric was cut into samples measuring four by twelve centimeters for experimental treatments according to ASTM D1682-64 Each sample was identified with a test detergent by corner marks One corner was clipped for samples treated with detergent one, two corners for detergent two, three corners for detergent three

Description of Samples

One hundred eight test fabric samples were used in the study Thirty-six samples were contaminated with the selected pesticide mixture and subjected to the experimental treatments, 36 samples were used as controls (blanks) and received experimental treatments but no pesticide contamination, and 36 samples were spikes and received contamination but no treatments It was necessary to establish the actual amount of pesticide applied to each sample for each contamination period since a percent was to be calculated and pesticides degrade over time For each replication four fabric samples, one for each detergent treatment and one spike sample, were pipette contaminated with 400 microliters of pesticide solution The spike sample was extracted and analyzed to establish the amount of contamination applied per fabric surface Because variability of measurement was introduced by both the gas chromatograph equipment and the injection syringe, each sample extractant was injected three times and the results averaged A summary of the average percent of pesticide recovered from the spike samples in Phase I and Phase II is given in Tables I and II

TABLE I

PHASE I PERCENT PESTICIDE RECOVERED FROM SPIKE SAMPLES

Spike Samples	Percent Recovery* Methyl Parathion	 Parathion
Weathered Samples		
Rep 1	100 0	100 1
Rep 2	100 6	97 6
Rep 3	92 7	106 7
Unweathered Samples		
Rep 1	100 0	93 9
Rep 2	97 0	97 0
Rep 3	86 3	97 9

*Represents an average of three injections per replication

TABLE II

PHASE II PERCENT PESTICIDE RECOVERED FROM SPIKE SAMPLES

Spike Samples	Percent Recovery*			
	Methyl Parathion		Parathion	
	Weathered	Unweathered	Weathered	Unweathered
Rep 1				
1	86 53	92 48	90 59	90 99
2	90 95	83 74	86 69	82 75
3	86 53	87 62	95 72	87 50
4	95 12	98 73	91 87	101 01
5	93 37	87 63	86 75	84 03
Rep 2				
1	91 35	91 15	92 42	70 39
2	87 01	99 18	88 84	78 59
3	86 30	80 90	88 22	83 70
4	86 20	80 90	90 04	71 50
5	64 54	87 10	64 17	74 56
Rep 3				
1	93 71	97 60	102 34	102 34
2	95 58	87 76	104 07	97 87
3	93 69	80 82	99 32	88 07
4	97 21	86 65	107 39	92 13
5	96 34	87 87	108 13	94 40
Mean	89 62	88 68	93 10	86 65

*Represents an average of three injections per replication

Because pesticides break down in the environment, there was concern that the pesticide could break down on the fabric samples during the drying time before the samples were subjected to the weathering and laundry treatments To determine whether the pesticide on the sample broke down during the one hour and forty-five minute drying time, three test fabric samples were contaminated and dried and analyzed with no further treatments Residue retention of these samples were subjectively compared to the residue recovered from the spike samples Due to variability introduced by the equipment and the researcher, three injections were made and averaged for each sample The mean percent of methyl parathion recovered from the three samples, measured by gas chromatograph, was 93 3 percent The mean percent recovered from the three samples for parathion was 97 8 The mean of the spike samples for Phase I was 96 1 percent for methyl parathion and 97 3 percent for parathion Parathion did not appear to break down during the drying period Methyl parathion may have degraded slightly

Description and Preparation of Pesticide

Pesticide

A commercial-grade emulsifiable concentrate pesticide, 6-3 Parathion-Methyl, formulated for the Platte Chemical Company was the selected pesticide for this study The active ingredients were parathion (O,O-diethyl o-p nitrophenyl Phosphorothioate) 55 3 percent, Related Compounds 2 6 percent, Methyl parathion (O,O-demethyl o-p-Nitrophenyl Phosphorothioate 27 5 percent, Related Compounds 1 4 percent and Inert Compounds 13 2 percent The E P A Reg No was 34704-16 (Restricted)

Pesticide Preparation

A series of steps were necessary to develop a homogeneous working solution from the water insoluble commercial grade methyl parathion and parathion compound Prior to beginning the study, a field strength solution (1 25%) was prepared Five hundred milliliters of working pesticide solution were prepared from 494 21 milliliters water and 5 79 milliliters of the commercial-grade pesticide The pesticide did not form a solution in the water but settled to the bottom of the container To promote solubility one percent acetone was added to the mixture

Further experimentation with the solution revealed that the mixture was an emulsion rather than a true solution Additionally, the emulsion contained tiny particles In an attempt to develop a more homogeneous working solution, the commercial-grade pesticide and pure nanograde quality acetone were combined to achieve a 1 25 percent field strength solution The acetone pesticide mixture was placed in an amber bottle, stirred with a magnetic stirrer and allowed to set overnight The floculent material settled out and was discarded after the supernatant was decanted The decanted mixture was analyzed by gas chromatograph and quantified for both parathion and methyl parathion

Because the chromatograph was not compatible with water, it was necessary to extract the water soluble acetone and pesticide into methylene chloride for analysis Fifty milliliters of methylene chloride, and ten milliliters of pesticide-water mixture were placed in a separatory funnel for the extraction process The mixture was allowed to set for ten minutes while the water rose to the top of the methylene chloride The methylene chloride was drained off and poured over sodium sulfate nested in a glass wool lined funnel to remove any traces of water The water and acetone in the flask were rinsed a second time with 20 milliliters methylene chloride The methylene chloride and extracted pesticide were placed in a Kuderna-Danish evaporative concentrator with floating glass valves (Micro Snyder Column) attached to a calibrated conical glass flask

The mixture was concentrated down to 10 milliliters in a water bath heated to 50°C The Kuderna-Danish was rinsed with two milliliters of methylene chloride followed with two milliliters of acetone The calibrated flask was removed, placed in a tube concentrator, and heated to 56°C to boil off the methylene chloride The remaining acetone and pesticide mixture were diluted in 10 milliliters of acetone Three one-microliter volumes were quantified by injection on the gas chromatograph

The methylene chloride and pesticide solution yielded a mean recovery of 620 µg/ml of methyl parathion and 971 µg/ml of parathion This was low compared to the recovery of 1696 µg/ml of methyl parathion and 2394 µg/ml of parathion of the decanted acetone solution Previous research has shown that heat contributes to the decay of pesticide (Sharom and Miles, 1981) Thus, the extraction process may have degraded some of the pesticide The water acetone working solution was finally quantified by direct injection on the gas chromatograph because the small quantity of water present in any one injection would be vaporized instantly by the high injection port temperature of the chromatograph

From the decanted mixture, 500 milliliters of pesticide solution was prepared to contaminate the test fabric specimens for this study The solution was prepared by mixing the decanted mixture with an equal volume of distilled water The solution was gas chromatograph analyzed to establish the average number of micrograms methyl parathion and parathion contained in one milliliter of the solution

The following procedure was used to determine the average micrograms per milliliter of parathion and methyl parathion contained in each milliliter of the solution Three (one milliliter) samples were removed from the prepared pesticide solution for analysis From each of the three one milliliter samples, three (one microliter) injections were drawn by syringe and injected on the chromatograph The results were averaged The average micrograms of methyl parathion and parathion present in each microliter of solution were established by comparison of gas chromatograph readings with one microliter pure standard of methyl parathion and parathion One gram of methyl parathion was obtained from EPA, and one gram of parathion was obtained from Allteck Chemical Company Each milliliter contained an average of 917 6 micrograms methyl parathion and 1382 0 micrograms parathion Four-tenths of a milliliter (400 microliters) were used to contaminate each test fabric sample Each 400 microliters of pesticide solution contained an average of 367 micrograms methyl parathion and 552 8 micrograms of parathion

Laundering and Weathering Equipment

Weathering Equipment

An Atlas Ci35 Fade-Ometer equipped with a xenon arc lamp was used to determine the effect of weathering on pesticide residue The test conditions used to simulate one day of weathering were temperature 63°C, humidity 65 percent, irradiance band 1 50 W/m^2, and test length three hours and forty minutes The test time was determined from data collected by the South Florida Testing Service (Scott, 1980) They determined that an irradiance level of 1144W/m^2 or (1357 hours) would be required to provide the equivalent energy of the average yearly irradiation received at the site of the South Florida Testing Service The three hour and forty minute test time was derived by dividing the 1357 hours per year by 365 days Thus, each weathering test period of three hours and forty minutes was the equivalent of one day of sunshine with the temperature at 63°C and a humidity level of 65 percent

The weathering time of one day was selected for practical purposes Protective clothing is expensive and pesticide handlers are not likely to own a quantity of protective garments It was reasonable to expect that handlers would have at least two garments and could allow one to hang for one day

Laundry Equipment

An Atlas Launder-Ometer equipped with 12 stainless steel canisters measuring 9 by 20 centimeters was selected Each canister lid was fitted with a neoprene gasket and teflon liner Fifty stainless steel balls in each canister, provided abrasive action, and the rotation of launder-ometer rack provided agitation

Detergents

The three detergents representing two surfactant types were selected for the three detergent treatments in this study Detergent one contained an anionic surfactant, detergent two contained a nonionic surfactant, and detergent three contained a combination of nonionic and anionic surfactants

The anionic type detergent was selected because the results of an on-going study of Oklahoman's with responsibility for laundering pesticide contaminated garments indicated that an anionic detergent was used most frequently for laundering The nonionic type detergent was chosen because it was chemically formulated to be particularly effective for oily soil removal (Soaps and Detergents, 1981) The detergent with combined anionic and nonionic surfactants was selected to capitalize on the positive features of both types of detergents

A description of the detergents is as follows Detergent one contained an anionic surfactant, water softeners (complex sodium phosphates, sodium carbonate), processing aids (sodium sulfate), washer protection agents, fabric whiteners, perfume, and agents to prevent deposition The phosphate content was 8 4 percent or the equivalent of 6 3 grams of phosphate per three-fourths cup of detergent Detergent two contained a nonionic surfactant, sodium tripolyphosphate, protease, and amylase This detergent contained a high phosphate content of 14 7 percent or the equivalent of 5 7 grams per one-fourth cup of detergent Detergent three contained anionic and nonionic surfactants, water softeners (complex sodium phosphate), processing aids (sodium sulfate), fabric whiteners, washer protection agent (sodium silicates), and perfume The Phosphate content was 8 0 percent or the equivalent of 5 8 grams per one-half cup of detergent

Sample Treatment Procedures

Contamination of Samples

Three fabric samples, one for each designated laundry detergent, were randomly placed on foil within a covered ventilated hood and contaminated Three control samples (blanks), one for each designated laundry treatment, received no contamination and were placed either in the fade-ometer to be weathered or in a launder-ometer canister to await laundering along with the contaminated samples An additional fabric sample was placed in a calibrated centrifuge tube for contamination as a spike Samples were contaminated with 400 microliters of pesticide solution with a Hamilton 200 microliter automatic inject pipette The spike sample was covered with ten milliliters of acetone, wrapped in aluminum foil and stored in the refrigerator The remaining samples were allowed to dry for one hour and forty-five minutes Dried samples were either subjected to simulated weathering or laundered immediately

Weathering of Samples

Thirty minutes prior to the start of the weathering test, the fade-ometer test conditions were set and the instrument was allowed to stabilize The dried contaminated samples and the three control samples were attached to fade-ometer sample holders with double stick tape to insure that no part of the surface of the fabric sample was covered by the holder Thus, the entire surface of sample was exposed to the xenon lamp The samples were placed only on the top portion of the fade-ometer rack for this test

Laundry Preparation

While the contaminated samples were drying, the launder-ometer and canisters were prepared for testing The canisters and lids were washed with Alconox laboratory detergent and rinsed with tap water, deionized water, and distilled water Both canisters and lids were labeled with tape to identify them with a contaminated sample or a control sample and an assigned detergent treatment One hour before beginning the laundry process, the launder-ometer tank was filled with tap water and allowed to heat to the specified water temperature of 49°C Fifty steel balls, 150 milliliters of room temperature deionized water, 0 3 milliliters detergent, and one fabric sample were placed in each canister Canister lids were fitted with new gaskets and teflon liners after each replication

Laundry Procedure

AATCC test method number 61-1980 (American Association of Textile Chemists and Colorists, 1984) was adapted to establish laundry procedures to simulate one home laundering The wash time of 45 minutes, as prescribed for AATCC test Method 61-1980, was reduced to 15 minutes Laughlin et al (1981) estimated that 15 minutes wash time in the launder-ometer was the equivalent of one home laundering The prepared canisters were placed in the launder-ometer rack three to a side When all 12 canisters were loaded the launder-ometer was operated for two minutes Then canister lids were released to equalize pressure and reclamped The launder-ometer was operated for 15 minutes to simulate one home laundering At the end of the wash cycle, the canisters were removed to the sink where the contents of each canister were poured into a strainer with a label matching the canister The fabric sample and the steel balls were returned to each canister in preparation for the rinse

Rinse Cycle

After completion of wash cycle, the temperature of the water in the launder-ometer tank was lowered to 40°C in preparation for the rinse cycle Samples received two rinses, one five-minute and one three-minute, each in 100 milliliters of 40°C deionized water

Drying

Wet samples were removed from the canisters with forceps and placed on layers of paper toweling with a foil underlay Samples were allowed to air dry for two hours before being stored to await extraction and analysis

Storage and Extraction

Dried samples were rolled up with forceps and put into 15 milliliter calibrated Kimbie bottles with screw tops Ten milliliters of nanograde acetone were added to each bottle to extract the pesticide Capped bottles were foil wrapped, shaken for 20 minutes on a Yankee Pipette Shaker to aid extraction, and stored in the refrigerator for 24 hours After 24 hours the fabric sample was removed from each bottle and discarded The bottles containing the extracted solution were rewrapped and placed in the refrigerator to await analysis by gas chromatograph

Acetone was used to extract pesticide contamination from the Gore-Tex® fabric samples To determine how well the acetone extracted the pesticide from the fabric samples, three fabric samples were placed in 15 milliliter bottles, and 400 microliters of the pesticide solution were pipetted onto each test fabric sample Ten milliliters of acetone were added to each bottle The bottles were foil wrapped and placed in the refrigerator for 24 hours After 24 hours the fabric samples were then discarded and the remaining acetone and pesticide solutions were gas chromatograph analyzed to determine the quantity of pesticide contained in each solution A 98 3 percent extraction efficiency was found

Gas Chromatograph

The extracted pesticide solution from the test fabric was analyzed by a Tracor 560 Gas Chromatograph with a flame ionization detector The chromatograph column was 5 percent OV-1 with mesh size of 80/100 on Supelcoport The settings were helium flow 3 0 pounds per square inch (psi), air 1 2 (psi), and hydrogen 30 0 (psi) The selected conditions were oven temperature 200°C, injection port 250°C, flame detector 230°C

Injections of three volumes of standard solutions of both methyl parathion and parathion were injected daily prior to running the samples The strength of both standards was 0 05 micrograms/microliter diluted in 10 milliliters of acetone A linear regression curve was established with a desired correlation coefficient of 0 995 or better Five microliters of the sample extracts, and three microliters of the spike extracts were injected with a Hamilton 10 0 microliter syringe

Procedure was replicated three times and the peak areas were averaged After every five samples, an injection of 1 0 microliter of the methyl parathion standard was made to check the consistency of chromatograph A new septa was inserted in inject port after every 30 injections At the end of each day's runs both the methyl parathion and parathion standards were reinjected

The gas chromatograph was connected to a Hewlett Packard 3990 Integrator recorder with settings of retention 0 00, chart speed 1 0 centimeters per minute, peak width 0 04, threshold 0, and area rejection 0 The integrator was programmed to compute peak retention time, and peak area A Texas Instruments Model 59 statistical calculator was programmed to convert peak areas into concentration of micrograms of pesticide injected on column, to determine the concentration of micrograms per microliter per injection of pesticide extract, and, to determine the micrograms of pesticide per fabric sample recovered

Statistical Analysis of Data

An analysis of variance (ANOVA) was used to determine the effect of detergent treatments and weathering for both Phase I and Phase II A separate statistical analysis was completed for methyl parathion and parathion A Duncan's multiple range post hoc test was used to distinguish between groups of significant means

CHAPTER IV

RESULTS AND DISCUSSION

The findings of this research are presented in three parts Phase I and Phase II results and discussion are given in Parts 1 and 2 respectively A comparison of Phase I and Phase II is the focus of the third part The purpose of Phase I was to determine the methyl parathion and parathion residue remaining in the Gore-Tex® test fabric and to determine the effects of detergent treatments and simulated weathering treatments Phase II determined whether repeated contaminations and subsequent simulated weathering and detergent treatments influenced pesticide residues remaining in the test fabric Pesticide residue build-up over time was also examined

Results of Phase I

Removal of Parathion

In the weathered samples the mean percentage of parathion residue retained after one laundering ranged from 31 91 to 38 02 percent (Table III) An examination of the means indicated that all of the detergents removed similar amounts of parathion The mean percentage of parathion retained in the unweathered samples after one laundering ranged from 40 09 to 46 16 percent A comparison of the weathered and unweathered sample means suggested that weathered samples

TABLE III

MEAN PERCENT PARATHION RETAINED AFTER ONE LAUNDERING

Variable	Weathered			Unweathered		
	Percent	µg/Sample	Std Dev	Percent	µg/Sample	Std Dev
Detergent 1	37 23	205 67	24 99	40 09	265 77	26 97
Detergent 2	31 91	178 03	23 74	43 80	245 17	30 44
Detergent 3	38 02	209 80	42 33	46 16	255 20	30 61
Grand Mean	35 72	197 83	-	43 08	255 38	-

retained less parathion after one laundering than did the unweathered samples

A 2x3 randomized block split plot analysis of variance was used to test the effect of weathering and detergent treatments on the removal of parathion from the test fabric An examination of the source table (Table IV) indicated that there was no significant interaction of weathering and detergent treatments (p< 05) Examination of the main effects (detergent treatments and weathering) indicated that detergent treatments were not significantly different (p<0 05) but the weathering treatment was significant (p< 04) The grand mean percent of parathion residue retained regardless of detergent treatment was 35 72 in the weathered samples and 43 08 in the unweathered samples (Table III) Thus, weathering did have a positive effect on the parathion removal from the test fabric samples

Removal of Methyl Parathion

The mean percentage of methyl parathion residue retained in the weathered samples after one laundering ranged from 7 41 to 22 44 percent (Table V) A visual examination of the weathered sample means revealed that detergent one, containing an anionic surfactant and detergent three, containing both anionic and nonionic surfactants were approximately equal in their effectiveness for removing methyl parathion Detergent two containing a nonionic surfactant appeared to perform better than the other two detergents In the unweathered samples the mean percentage of methyl parathion residue retained after one laundering ranged from 22 04 to 24 55 percent (Table V) An examination of the means indicated that all three detergents were similarly effective in

TABLE IV

ANALYSIS OF VARIANCE TABLE - WEATHERING AND DETERGENTS FOR PARATHION AFTER ONE LAUNDERING

Source	Sum of Squares	Degrees of Freedom	Mean Square	F Ratio
Total	33472 13	17		
Between Weathering	22545 88	5		
Block	6453 31	2	3226 65	5 42
Weathering	14901 13	1	14901 13	25 01*
Error (Weathering)	1191 44	2	595 72	
Within Weathering	10926 25	12		
Treatments	2057 54	2	1028 77	0 97
Weathering (Treatments)	358 94	2	197 47	0 17
Error (Treatments)	8499 75	8	1062 47	

*Significant at the 05 level

TABLE V

MEAN PERCENT METHYL PARATHION RETAINED AFTER ONE LAUNDERING

Variable	Weathered			Unweathered		
	Percent	µg/Sample	Std Dev	Percent	µg/Sample	Std Dev
Detergent 1	19 30	70 83	22 83	23 54	86 43	20 37
Detergent 2	7 41	40 80	17 84	22 04	85 47	35 05
Detergent 3	22 44	81 90	32 65	24 55	90 10	27 55
Grand Mean	16 38	64 51	-	23 37	81 33	-

removing methyl parathion from the test fabric A comparison of the weathered and unweathered sample means indicated that all of the weathered samples regardless of detergent treatment retained less methyl parathion residue than did the unweathered samples Thus, it appeared that weathering aided in methyl parathion removal from the test fabric

A 2x3 randomized block split plot analysis of variance was used to test the effect of weathering and detergent treatments on the removal of methyl parathion from the test fabric An examination of the data in Table VI indicated there was no significant interaction of weathering and laundry treatments (p< 05) An examination of the main effects (detergent treatments and weathering) indicated that the detergent treatments were not significantly different (p< 05) Weathering was significant (p< 03) The grand mean percent of methyl parathion residue retained, regardless of detergent, was 16 38 percent in the weathered samples and 23 37 percent in the unweathered samples (Table V) Thus, there was a consistent pattern for the weathered samples to retain less methyl parathion and parathion residue than the unweathered samples

Additionally, an examination of the data presented in Table VI indicated a significant block effect at the 0 01 level The significant block effect may be attributed to a nonhomogeneous solution of the pesticide used for contaminating the samples The pesticide was a mixture containing parathion and methyl parathion to which water was added to make it a field strength concentration Acetone was added to improve the likelihood of the pesticide staying in a homogeneous solution Despite this effort, the methyl parathion may not have been homogeneous from day to day as each replication was completed

TABLE VI

ANALYSIS OF VARIANCE TABLE - WEATHERING AND DETERGENTS FOR METHYL PARATHION AFTER ONE LAUNDERING

Source	Sum of Squares	Degrees of Freedom	Mean Square	F Ratio
Total	18079 80	17		
Between Weathering	12535 13	5		
Block	10048 69	2	5024 35	69 91*
Weathering	2342 70	1	2342 70	32 60**
Error (Weathering)	143 74	2	71 87	
Within Weathering	5544 69	12		
Treatments	1635 41	2	817 71	2 34
Weathering (Treatments)	1115 46	2	557 73	1 60
Error (Treatments)	2793 82	8	349 23	

*Significant at the 01 level

**Significant at the 05 level

Summary-Discussion of Phase I Findings

Hypotheses one and two failed to be rejected Thus, detergent treatments did not significantly influence pesticide removal from the test fabric Hypotheses three and four were rejected, the weathering treatment significantly reduced the parathion and methyl parathion residue retained in the test fabrics

Weathering Effect

Samples that were subjected to simulated weathering retained significantly lower amounts of methyl parathion and parathion residue It is not known whether any one of the weathering components was more influential on the break down of the pesticide from the test fabric This finding agrees with pesticide degradation studies which found that pesticides broke down in the natural environment due to the interaction of heat, light, humidity, organic and inorganic substances over time No research on the degradation of pesticides in fabric have been found However, pesticide degradation studies using other products found that pesticides do break down over time in the presence of a variety of mediums Davies et al (1977) found that concentrated methyl parathion spilled on cement blocks and exposed to sunlight, natural heat, and moisture decayed after three months exposure, while methyl parathion spilled on cement blocks and stored indoors free from sunlight and moisture took four and one-half months to decay Thus, the sunlight, moisture, and temperature effect on the samples stored outdoors appeared to accelerate the decay of the pesticide Sharom and Miles (1981) found that parathion, added to water, completely disappeared after being stored at room temperature for two weeks Kawar, Gunther, and Iwata (1978) also determined that parathion laden wine contained significantly lower levels of parathion after two months of storage

Draper and Street (1981) found that methyl parathion applied to alfalfa in Utah during July (sunlight, temperature, and humidity unknown) decayed significantly within 28 hours after application Wolfe et al (1975) found that parathion applied to apple orchards in Washington in June (sunlight, temperature, and humidity unknown) decreased by approximately 50 percent within 24 hours after application Thus, previous research indicates that the time required for parathion and methyl parathion to decay varies from a few hours to several months Decay may be related to a complex interaction of variables with the pesticide Which variable (heat, sunlight, humidity) contributed most to pesticide break down is unknown

Contaminated fabric samples that received the weathering treatments in the fade-ometer had brown stains after one laundry cycle Whether the stains were residues of the pesticide or some nonactive ingredient in the carrier solution of the commercial-grade pesticide solution is unclear However, there is indication that one or more of the weathering variables of heat, light, or humidity interacted to form the stains as no staining was observed in any of the contaminated samples that did not receive weathering treatments

Detergent Effect

High percentages of both methyl parathion and parathion residues were retained in the Gore-Tex® regardless of the detergent used Fiber content may have contributed to pesticide residue retention in the Gore-Tex® The nylon components of the Gore-Tex® fabric were hydrophobic in nature Thus, the Gore-Tex® fabric was difficult to wet in an aqueous medium Bowers and Chantrey (1969) found that the hydrophobicity of a fabric was related to ease of soil removal Those that were difficult to wet with water were difficult to clean using an aqueous laundry liquor The nylon fiber of the Gore-Tex®, in addition to being hydrophobic, had an oleophilic property While the fiber repelled water, it had the potential to absorb the oily pesticide Easter (1982) likewise, found that fabrics composed of synthetic fibers were more difficult to decontaminate of Guthion® and captan than fabrics containing cotton fibers Despite these studies indicating greater difficulty in removing pesticides from synthetic fabrics, Obendorf, Namaste, and Durnam (1983), and Solbert and Obendorf (1985) did not find malathion and methyl parathion absorbed into the interior of nylon, polyester, or acrylic Rather they found a thin oily film uniformly distributed on the exterior of the fibers Further research is needed to better understand the mechanisms at work

Additionally, factors other than the oleophilic properties of synthetic fabrics may have contributed to pesticide retention in the Gore-Tex® Bowers and Chantrey (1969) found that fabric construction influenced dislodgement of oily soil by inhibiting circulation of detergent solution through the interfiber capillary system formed by the close spaces between the fine filament fibers Brown, Thompson, and Stewart (1968) determined that residual oily soil was predominately located on fiber surfaces and in the interfiber spaces of the yarns Bowers and Chantrey (1969) found that a tightly woven fabric with high twist yarns was more difficult to clean than a loosely woven fabric with low twist yarns Thus, the construction of Gore-Tex®, a tightly woven nylon rip-stop laminated to the impermeable membrane, may have contributed to the high residue retention because the laundry liquor could not circulate through the fabric during the laundry process to dislodge and carry away the pesticide residue

The pesticide residues retained in the Gore-Tex® ranged from 7 41 to 46 16 percent However, the quantity of the pesticide available for dermal absorption is unknown The pesticide contamination was applied to the layer of the Gore-Tex® that would be worn away from the skin in a protective garment If the pesticide residue remained on the surface layer of the Gore-Tex® after laundering, the impermeable microporous film of the Gore-Tex® would prevent the pesticide from dermal contact If, however, the pesticide transferred into the laundry liquor and re-deposited on the uncontaminated layer of Gore-Tex®, a potential health hazard may occur Whether the residue retained in the Gore-Tex® bonded to the synthetic fibers as a result of treatments or whether it could transfer after laundering to the skin is unknown Thus, additional investigation to determine the precise location and availability of the pesticide for dermal transfer is necessary before Gore-Tex® could be recommended as a viable fabric for use in protective garments

Results of Phase II

Removal of Repeat Contamination of Parathion

The mean micrograms per sample of parathion residue retained in the weathered samples, five times contaminated and laundered, ranged from 154 65 µg to 220 07 µg (Table VII) An examination of the means of the weathered samples revealed that detergents two and three were more

TABLE VII

MEAN PARATHION RESIDUE RETAINED AFTER FIVE LAUNDERINGS

Variable	Weathered		Unweathered	
	μg/Sample	Std Dev	μg/Sample	Std Dev
Detergent 1	220.07	11.71	312.23	22.64
Detergent 2	159.93	8.44	195.34	17.69
Detergent 3	154.65	40.98	292.19	11.33
Grand Mean	178.22	-	266.59	-

effective than detergent one in removing parathion from the test fabric Parathion, measured in micrograms per sample, retained in the unweathered samples ranged from 195 34 µg to 312 23 µg An examination of the data in Table VII shows that detergent two removed parathion better than detergents one and three The grand mean parathion residue retained, regardless of detergent treatment in the weathered samples was 178 22 µg as compared to 266 59 µg in the unweathered samples (Table VII)

A 2x3 randomized split plot analysis of variance was used to test the effect of weathering and detergent treatments on the removal of parathion from the test samples An examination of the data in Table VIII indicated a significant interaction of weathering and detergent (p< 02) The combination of detergent two and weathering yielded the least amount of parathion residue It should also be noted that variability was a problem, particularly with detergent three weathered samples Also, detergent was significant (p< 01) and weathering was significant (p< 04), with the weathered samples retaining less parathion than the unweathered samples A Duncan's multiple range post-hoc test (Table IX) showed that the means of the unweathered samples laundered in detergents one and three were not significantly different (p< 05) from each other but were significantly different from all other means The mean of the weathered sample laundered in detergent one and the unweathered sample laundered in detergent two were not significantly different (p< 05) from each other The means of the weathered samples laundered in detergents two and three and the mean from the unweathered sample laundered in detergent two were not significantly different (p< 05) from each other Those means designated in Table IX with the letter C reflect the least amount of residue retained

TABLE VIII

ANALYSIS OF VARIANCE TABLE - WEATHERING AND DETERGENTS FOR PARATHION AFTER FIVE LAUNDERINGS

Source	Sum of Squares	Degrees of Freedom	Mean Square	F Ratio
Total	75036 01	17		
Between Weathering	39499 91	5		
Block	1854 82	2	927 41	
Weathering	35144 31	1	35144 31	28 11*
Error (Weathering)	2500 78	2	1250 39	
Within Weathering	35536 10	12		
Treatments	23513 15	2	11756 58	22 58*
Weathering (Treatments)	7856 81	2	3928 41	7 54*
Error (Treatments)	4166 14	8	520 76	

*Significant at the 05 level

TABLE IX

DUNCAN'S MULTIPLE RANGE TEST FOR PARATHION RESIDUE BY DETERGENTS AFTER FIVE LAUNDERINGS

Variable	Mean µg/Sample	Duncan's Groupings[a]
Detergent 1 (Unweathered)	312 23	A
Detergent 3 (Unweathered)	292 19	A
Detergent 1 (Weathered)	220 07	B
Detergent 2 (Unweathered)	195 34	B C
Detergent 2 (Weathered)	159 93	C
Detergent 3 (Weathered)	154 65	C

[a] Means with the same letter are not significantly different

Removal of Repeat Contamination of Methyl Parathion

The mean micrograms per sample of methyl parathion residue retained in the weathered samples repeatedly contaminated and laundered ranged from 51 7 μg to 74 58 μg (Table X) A visual examination of the means of the weathered samples suggests that detergent two was more effective in removing methyl parathion than detergents one and three Methyl parathion residue, measured in micrograms per sample, remaining in the unweathered samples, after five consecutive contaminations and launderings, ranged from 62 34 μg to 100 09 μg (Table X) A similar pattern for more effective methyl parathion removal by detergent two was observed with the unweathered test fabric samples The grand mean methyl parathion residue retained, regardless of detergent treatment, in the weathered samples was 65 60 μg as compared to 83 96 μg in the unweathered samples Thus, weathering consistently showed a positive effect on the removal of methyl parathion from the test fabric samples

A 2x3 randomized block split plot analysis of variance was used to test the effect of weathering and detergent treatments on the removal of methyl parathion from the test samples An examination of the data in Table XI indicated no significant interaction between weathering and detergent treatments Therefore, the main effects of weathering and detergents were examined Weathering was not significant ($p <$ 05) A possible explanation for the weathering not being significant may be attributed to the construction of the Gore-Tex® fabric Because contamination was repeated, the pesticide may have penetrated beneath the surface of the first fabric layer where the light of the fade-ometer would not be available to break down the chemical The detergent

TABLE X

MEAN METHYL PARATHION RESIDUE RETAINED AFTER FIVE LAUNDERINGS

Variable	Weathered		Unweathered	
	µg/Sample	Std Dev	µg/Sample	Std Dev
Detergent 1	74 58	5 72	89 47	11 28
Detergent 2	51 71	5 59	62 34	10 38
Detergent 3	70 58	5 37	100 09	14 22
Grand Mean	65 60		83 96	

TABLE XI

ANALYSIS OF VARIANCE TABLE - WEATHERING AND DETERGENTS FOR METHYL PARATHION AFTER FIVE LAUNDERINGS

Source	Sum of Squares	Degrees of Freedom	Mean Square	F Ratio
Total	6267 98	17		
Between Weathering	2682 74	5		
Block	584 17	2	292 09	0 98
Weathering	1499 51	1	1499 51	5 01
Error (Weathering)	599 06	2	299 53	
Within Weathering	3585 25	12		
Treatments	2892 12	2	1446 06	28 35*
Weathering (Treatments)	285 08	2	142 54	2 79
Error (Treatments)	408 04	8	51 01	

*Significant at the 01 level

treatment was significant ($p < 05$) These findings conflict with the findings of Phase I, where after one laundering, weathering did significantly affect methyl parathion removal and detergent treatments did not

One possible explanation for the significant detergent effect found in Phase II might be attributed to the nonionic surfactant's affinity for removal of oily soils According to Soaps and Detergents (1974), the nonionic type detergents should be particularly effective for removing oily type soils because of their chemical composition Typically, surfactants have two parts a hydrophobic part and a hydrophilic part The hydrophobic part has an affinity for oil while the hydrophilic part has an affinity for water Thus, the opposite forces imposed by the two parts of the surfactants pull the oily soil from the fabric surface and suspend it in the wash water until it is carried away The nonionic type surfactant has a larger hydrophilic part and more power to pull oily soil from the fabric Perhaps repeated launderings were necessary before a trend for more effective performance by detergent two became significant An examination of the data in Table VII indicated that, with the exception of the weathered parathion contaminated samples laundered in detergent three, there was a consistent pattern for detergent two to perform more effectively than detergents one and three

The more efficient performance of detergent three in the weathered samples might be attributed to the large variability within the samples laundered in detergent three When the gas chromatograph analysis of the second replication of samples, laundered in detergent three, was in progress the air supply to the gas chromatograph diminished due to low volume of air in the supply tank When the air supply was restored the gas chromatograph equipment registered an increased sensitivity for measuring parathion which resulted in marked variability in the data

Summary-Discussion of Phase II Findings

Hypothesis five was rejected Detergent treatments were not significantly different for removing parathion residues from the test fabric Hypothesis six failed to be rejected Thus, detergent treatments were significantly different for removing methyl parathion residues from the test fabric Hypothesis seven failed to be rejected Thus, weathering treatments did significantly reduce parathion residues in the test fabric Hypothesis eight was rejected Weathering treatments did not significantly reduce methyl parathion in the test fabric

Parathion Removal

There was a consistent pattern for the weathered samples five times contaminated and laundered to retain less parathion residue than the unweathered samples Detergent two performed more effectively than other detergents tested for removing parathion from the test fabric in the unweathered samples In the weathered samples detergent two did not perform more effectively than detergent three, but both performed more effectively than detergent one

Methyl Parathion Removal

Weathering was not significant for removal of methyl parathion in five times contaminated and laundered samples While weathering was not significant, there was a consistent trend for the weathered samples to retain less methyl parathion Variability in the study could be responsible for differences in weathering effects between Phase I and II Further research with a larger number of samples and an increase in the number of replications would be necessary before any definite conclusions could be drawn regarding the effect of weathering on the break down of methyl parathion in Gore-Tex® fabric

There was a significant detergent effect for removal of methyl parathion An examination of the means of the methyl parathion residue (Table X) indicated that detergent two was more effective for methyl parathion removal than was either of the other two detergents As was explained previously, the nonionic surfactant with an affinity for removing oily soils may have been responsible for the more effective performance Fabric, fiber, and pesticide characteristics included in the discussion of Phase I would also apply to Phase II

Comparison of Phase I and Phase II Findings

Though not statistically tested, a subjective comparison of the results of Phase I and Phase II was made to determine whether pesticide residue built-up in samples repeatedly contaminated and subjected to the weathering and detergent treatments The grand mean parathion residue retained in weathered samples of Phase I was 197 83 µg as compared to the grand mean of 178 22 µg in Phase II samples (Table XII) Observation of the grand means indicated a small difference between the residue retained in the repeatedly contaminated and laundered samples subjected to weathering and one time contamination and laundering samples However, an observation of the grand means of unweathered

TABLE XII

COMPARISON OF GRAND MEANS OF PHASE I AND PHASE II FOR PARATHION

Samples	Phase I		Phase II
	Percent	µg	µg
Weathered	35 72	197 83	178 22
Unweathered	43 35	255 38	266 59

parathion residue retained in Phase I was 255 38 µg per sample compared to 266 59 µg per sample in Phase II (Table XII) Thus, in the unweathered samples no evidence of a build-up of parathion was apparent

The grand mean methyl parathion residue retained in the weathered samples of Phase I was 64 51 µg (Table XIII) as compared to 65 60 µg residue retained in samples from Phase II These observations indicated no build-up of methyl parathion in repeatedly contaminated, laundered, and weathered samples In the unweathered methyl parathion contaminated samples, the Phase I grand mean residue retained was 87 33 µg compared to 83 96 µg for the grand mean of Phase II (Table XIII) Thus, again there was no evidence of build-up after repeated contaminations and laundering

Comparison of Removal of Parathion and Methyl Parathion

Because both methyl parathion and parathion were contained in the commercial grade pesticide used in this study, no statistical test could be made comparing residue retention due to pesticide However, subjective observations were made of the percents of methyl parathion and parathion retained in Phase I The mean percent of pesticide retained in the weathered samples was 16 38 percent for methyl parathion and 35 72 percent for parathion In the unweathered samples the grand mean percent methyl parathion was 23 37 percent and 43 35 percent in unweathered parathion samples Thus, it appears that parathion was more difficult to decontaminate from Gore-Tex® than was the methyl parathion

TABLE XIII

COMPARISON OF GRAND MEANS OF PHASE I AND PHASE II FOR METHYL PARATHION

Samples	Phase I		Phase II
	Percent	µg	µg
Weathered	16 38	64 51	65 60
Unweathered	23 37	87 33	83 96

CHAPTER V

SUMMARY, IMPLICATIONS, AND RECOMMENDATIONS

Increased use of pesticides in all segments of the agricultural industry have resulted in a growing potential for pesticide handlers to experience dermal exposure Because absorption of pesticides through the skin may be hazardous to health, there is a need for comfortable and cleanable protective garments to guard against exposure

Summary of Equipment and Procedures

The purpose of Phase I was to determine the methyl parathion and parathion residue remaining in the Gore-Tex® test fabric and to determine the effects of detergent treatments and simulated weathering treatments Phase II determined whether repeated contaminations and subsequent simulated weathering and detergent treatments influenced pesticide residue remaining in the test fabric Pesticide residue build-up over time was also examined

A commercial-grade organophosphate pesticide containing both methyl parathion and parathion was selected for this research A working solution containing an average of 917 micrograms of methyl parathion and 1382 micrograms of parathion per microliter was mixed with 50 percent distilled water and 50 percent nanograde acetone Four hundred microliters of the solution was pipetted onto the surface of each test fabric sample Each 400 microliters contained 367 micrograms of methyl parathion and 552 8 micrograms of parathion

One test fabric, Gore-Tex® was selected Prior research identified Gore-Tex® as impermeable to selected tested pesticides and having a similar thermal comfort level of cotton chambray Gore-Tex® was a three layered fabric with an outer layer of rip-stop nylon, an inner layer of nylon tricot, and a microporous film of polytetrafluorecethylene laminated to the two other fabric layers

Half of the contaminated samples were subjected to simulated weathering conditions in an Atlas Ci35 Fade-Ometer equipped with a xenon arc lamp before being laundered Weathering conditions selected were temperature 63°C, humidity 65 percent, irradiance band of 1 50 w/m^2, and conditioning time three hours and forty minutes The other half of the contaminated samples received only laundry treatments

The samples receiving only laundry treatments and the weathered samples were laundered at the same time in an Atlas Launder-Ometer equipped with 12 stainless steel canisters fitted with teflon-lined lids In addition to a fabric sample, each canister contained 50 steel balls, 200 milliliters of deionized water, and 0 3 milliliters detergent Wash water temperature was 49°C and the rinse water temperature was 40°C

Three phosphate detergents representing two surfactant types were chosen Detergent one contained an anionic surfactant, detergent two contained a nonionic surfactant, and detergent three contained both an anionic and a nonionic surfactant

Laundered samples were placed in 15 millimeter glass calibrated bottles and covered with 10 milliliters of acetone Bottled samples were wrapped in aluminum foil, shaken 15 minutes on a pipette shaker, then stored in a refrigerator 24 hours to self-extract before being removed and discarded The extract was analyzed for pesticide residues by injection on a gas chromatograph equipped with a flame ionization detector

Methyl parathion and parathion were analyzed separately using a 2x3 randomized block split plot analysis of variance to test the effect of weathering and detergent treatments on the pesticide residue retained in laundered test fabric samples

Summary of Findings

Phase I

The grand mean of percent parathion residue retained in the weathered and laundered samples was 35 72 percent compared to 43 08 percent in the unweathered and laundered samples An analysis of variance indicated weathering was significant ($p<$ 05) There was no significant difference in the performance of the three detergents

The grand mean percent methyl parathion residue retained in the weathered and laundered samples was 16 38 percent compared to 23 37 percent in the unweathered and laundered samples An analysis of variance indicated weathering was significant at the 05 level There was no significant difference in the performance of the three detergents

Phase II

The weathered samples repeatedly parathion contaminated and laundered retained mean residues of 178 22 µg per sample compared to

266 59 μg in the unweathered samples An analysis of variance indicated a significant interaction of weathering and detergent treatments (p< 02) A combination of detergent two and weathering yielded the least amount of parathion residue The main effect, weathering treatments, was significant (p< 04) The weathered samples retained less parathion than the unweathered samples Detergent treatments were significant (p< 01) Detergent two appeared to be the most effective in parathion removal However, a large amount of variability was observed, particularly for detergent three This may have been due to difficulties in maintaining a homogeneous pesticide solution

The weathered samples repeatedly methyl parathion contaminated and laundered retained mean residues of 65 60 μg compared to 83 96 μg in the unweathered samples An analysis of variance indicated weathering was not significant (p< 05) Though not significant, there was a pattern for the weathered samples to retain less residue There was a significant difference in the performance of the three detergents (p< 05) Detergent two performed more effectively than either detergent one or three for removing methyl parathion from both the weathered and unweathered samples The mean micrograms per sample of methyl parathion retained by samples laundered in detergent two was 51 71 μg as compared to 74 58 μg for detergent one and 70 58 μg for detergent three

The results of Phase I and Phase II are conflicting as to the significance of weathering on the retention of methyl parathion While weathering was not significant, there was a consistent trend for the weathered samples to retain less methyl parathion residue than the unweathered samples Likewise, there was a conflict as to the significance of detergent performance between the two phases There was no significant difference in the performance of the three detergents in Phase I while there was a significant difference in performance in Phase II Because of variability in the study, conclusions regarding detergent performance on pesticide removal from the test fabric are inconclusive

A subjective comparison of the results of Phase I and Phase II was made to determine whether pesticide residue built-up in samples repeatedly contaminated and laundered In Phase I the grand mean residue retained in the weathered samples contaminated with methyl parathion was 64 51 µg as compared to 65 60 µg residue retained in samples from Phase II These observations indicated no build-up of methyl parathion in repeatedly contaminated and laundered samples In the unweathered methyl parathion contaminated samples, the Phase I grand mean residue retained was 87 33 µg compared to 83 96 µg for the grand mean of Phase II Thus, again there appeared no evidence of build-up after repeated contaminations and laundering

The grand mean parathion residue retained in weathered samples of Phase I was 197 83 µg as compared to the grand mean of 178 22 µg in Phase II samples Observation of the grand means indicated a small difference between the residue retained in the repeatedly contaminated, laundered, and weathered samples and the one time contamination, laundered, and weathered samples An observation of the grand means of the unweathered parathion residue retained in Phase I was 266 59 µg per sample compared to 255 38 µg per sample in Phase II Thus, in the unweathered samples, no evidence was apparent of a build-up of parathion

Because both methyl parathion and parathion were contained in the commercial-grade pesticide used in this study, no statistical test could be made comparing residue retention due to pesticide However, subjective observations were made of the percents of methyl parathion and parathion retained in Phase I The grand mean percent of methyl parathion in the weathered samples was 16 38 percent and 35 72 percent in weathered parathion samples In the unweathered samples the grand mean percent of methyl parathion was 23 37 percent and 43 35 percent in unweathered parathion samples Thus, it appears that parathion was more difficult to decontaminate from Gore-Tex® than was the methyl parathion

Implications of Results

Methyl parathion and parathion residues retained in weathered samples of Phase I were significantly lower than in the unweathered samples However, in Phase II the effect of weathering on retention of both pesticides was only significant for parathion There was, however, a consistent pattern for the weathered samples to retain less parathion and methyl parathion than did the unweathered samples After five contaminations and launderings, there was no clear evidence that weathering was effective in breaking down the pesticide prior to laundering Thus, the results are inconclusive

The contaminated samples subjected to simulated weathering developed stains while the unweathered samples did not Thus, if Gore-Tex® were made into protective garments and worn out in the sunlight, staining could be a problem

There is no clear indication whether one type of detergent could be recommended over another In some instances, detergent two appears to perform better than the other detergents tested This detergent had a nonionic surfactant but also a higher phosphate content Additional research is needed to better understand the role of detergents in the pesticide decontamination process

Recommendations for Research

1 Further research is needed to determine whether weathering is an effective means of breaking down pesticide prior to laundering

2 Further research of contaminated and weathered samples is needed to investigate the nature and location of stains within the fabric structure, and to determine effective means of removal

3 Further investigation is necessary to determine the level at which residue within fabrics becomes a health hazard

4 Further investigation is needed to determine whether pesticide residue remained on the surface of the Gore-Tex® or whether it transferred to the other side during the laundry process

5 Further investigation is needed to determine whether repeat launderings would reduce the amount of residue retained in the Gore-Tex®

6 Further investigation is needed to determine whether longer periods of weathering are necessary to break down the methyl parathion and parathion

7 Further research is needed to determine whether increasing the amount of laundry detergent per laundering would remove greater amounts of both pesticides

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