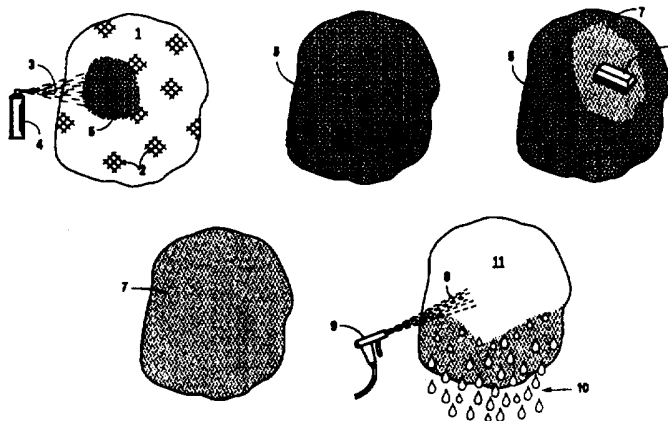




## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<p>(51) International Patent Classification<sup>6</sup> : C11D 3/14, 3/37, 3/43, 3/44, 3/48, 7/50, 7/60, C23G 1/00, 5/024</p>	A1	<p>(11) International Publication Number: <b>WO 96/20995</b></p> <p>(43) International Publication Date: 11 July 1996 (11.07.96)</p>
<p>(21) International Application Number: PCT/US96/00208</p> <p>(22) International Filing Date: 5 January 1996 (05.01.96)</p> <p>(30) Priority Data: 08/369,548 6 January 1995 (06.01.95) US</p> <p>(71) Applicant (for all designated States except US): TEXAS RESEARCH INSTITUTE [US/US]; 9063 Bee Caves Road, Austin, TX 78733-6201 (US).</p> <p>(72) Inventors; and (75) Inventors/Applicants (for US only): DINGUS, Michael, L. [US/US]; 1515 Oxford Avenue, Austin, TX 78704 (US). ZOCH, Walter, P. [US/US]; 11 Tall Oaks Trail, Austin, TX 78737 (US). MAYFIELD, Thomas, R. [US/US]; 1715 Ben Crenshaw Way, Austin, TX 78746 (US). BRAY, Alan [US/US]; 113 Cuernavaca Drive, Austin, TX 78733 (US). RUSHING, Rock, A. [US/US]; P.O. Box 161752, Austin, TX 78716 (US).</p> <p>(74) Agent: MAYFIELD, Denise, L.; Akin, Gump, Strauss, Hauer &amp; Feld, L.L.P., Suite 1900, 816 Congress Avenue, Austin, TX 78701 (US).</p>	<p>(81) Designated States: CA, JP, US, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).</p> <p>Published With international search report.</p>	

(54) Title: COMPOSITIONS CONTAINING A VISIBLE COLORANT AND METHODS FOR CLEANING AND DECONTAMINATION



## (57) Abstract

Improved compositions containing a visible coloring agent, such as a colored pigment or dye, together with a polymer or hydroxylated aliphate alcohol, a surfactant and in some embodiments, a therapeutic agent or biocide, are disclosed. The detectable coloring agent is readily visible under normal white light, and provides a technique for monitoring disturbed and undisturbed areas on a surface. The visible coloring agent of the compositions are further defined as imparting a color to the composition that remains virtually constant upon contact with a contaminant or change in pH of the composition, or of the surface or other environment to which it is exposed. Such is useful in the described methods for cleaning and/or decontaminating a surface, as in the decontamination of equipment and clothing used during hazardous spill response. The compositions are adherent to a variety of different materials, including Teflon®. This makes the preparations particularly useful in the cleaning and decontamination of non-flat and curved surfaces, such as on protective garments. The compositions, in particular embodiments, include a visually detectable coloring agent (such as a colored pigment), a surfactant (such as ethoxylate alcohol), and emulsifier (such as carboxymethyl cellulose), an extender (such as PEG), and a solvent (such as d-limonene). These compositions may also include water or other suitable diluent.

**FOR THE PURPOSES OF INFORMATION ONLY**

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AM	Armenia	GB	United Kingdom	MW	Malawi
AT	Austria	GE	Georgia	MX	Mexico
AU	Australia	GN	Guinea	NE	Niger
BB	Barbados	GR	Greece	NL	Netherlands
BE	Belgium	HU	Hungary	NO	Norway
BF	Burkina Faso	IE	Ireland	NZ	New Zealand
BG	Bulgaria	IT	Italy	PL	Poland
BJ	Benin	JP	Japan	PT	Portugal
BR	Brazil	KE	Kenya	RO	Romania
BY	Belarus	KG	Kyrgystan	RU	Russian Federation
CA	Canada	KP	Democratic People's Republic of Korea	SD	Sudan
CF	Central African Republic	KR	Republic of Korea	SE	Sweden
CG	Congo	KZ	Kazakhstan	SG	Singapore
CH	Switzerland	LI	Liechtenstein	SI	Slovenia
CI	Côte d'Ivoire	LK	Sri Lanka	SK	Slovakia
CM	Cameroon	LR	Liberia	SN	Senegal
CN	China	LT	Lithuania	SZ	Swaziland
CS	Czechoslovakia	LU	Luxembourg	TD	Chad
CZ	Czech Republic	LV	Larvia	TG	Togo
DE	Germany	MC	Monaco	TJ	Tajikistan
DK	Denmark	MD	Republic of Moldova	TT	Trinidad and Tobago
EE	Estonia	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	UG	Uganda
FI	Finland	MN	Mongolia	US	United States of America
FR	France	MR	Mauritania	UZ	Uzbekistan
GA	Gabon			VN	Viet Nam

## COMPOSITIONS CONTAINING A VISIBLE COLORANT AND METHODS FOR CLEANING AND DECONTAMINATION

The present application is a continuation-in-part of co-pending U.S. Patent Application Serial No. 08/369,548, filed January 6, 1995. The entire text of the above-referenced disclosure is specifically incorporated by reference herein without disclaimer. The government may own rights in the present invention as research was supported by U.S. Army Grant Number: DAAH04-93-C-0012.

### BACKGROUND OF THE INVENTION

#### 1. Field of the Invention

The invention in general relates to the **field** of cleaning and decontamination products and methods. More particularly, the invention relates to methods of increasing washing effectiveness, decontamination, cleaning, or disinfection of personal protective clothing, personnel, and equipment.

#### 2. Description of the Related Art

The release of hazardous and potentially toxic substances is a problem that requires prompt and effective treatment. Methods for handling release of such materials have been developed by the Environmental Protection Agency, (EPO Handbook, "The Standard Operating Safety Guidelines" (1984)). This handbook, and regulations promulgated by the Environmental Protection Agency's Occupational Health and Safety Manual (Chapter 9, Hazardous Substances Responses (1440 TN 12) (May 5, 1984)), also describe precautionary measures currently employed focussed at minimizing contamination to the environment and personnel involved in hazardous material cleanup and disposal.

Personal protective clothing, equipment, sampling tools and other items are also exposed to contaminating materials during clean-up and first responder situations. Garments that require cleaning in such applications include total encapsulating vapor protective suits as well as other equipment and protective coverings. These items must be cleaned and/or decontaminated as thoroughly as possible to minimize cross-contamination, and, in some cases, to prepare them for reuse. The cleaning and/or decontamination procedure should also be completed as quickly as possible. This is because workers in contaminated areas typically wear Self Contained Breathing Apparatus (SCBA) equipment. As such, the wearer is frequently low on air, thus requiring that

personnel be removed from the suit as soon after the hazardous task has been completed as possible.

5           Currently proposed techniques for removing contaminants from a surface may be classified as either physical or chemical. An example of a simple physical technique involves a detergent having an anionic surface active agent. One such detergent is characterized as having a boiling point of 212° F and a freezing point of 32° F. This detergent is non-adherent. Therefore, areas that have been washed with the detergent in the cleaning/decontamination process are not easily detectable. For water soluble substances, the cleaning procedure may simply amount to a detergent and water wash. 10 For acids and bases, neutralization employs a weak water-based solution of the opposing acid or base (e.g., vinegar and baking soda). Some acids, such as hydrofluoric acid, may need to be complexed as well as neutralized, in the decontamination process. Unlike the nuclear industry, use of solvents such as Freon® or methylene chloride has not been shown to be effective in the cleaning of organic substances, and may actually degrade or permeate the surface of a piece of equipment. One approach for cleaning volatile organics, as well as chemical warfare agents, is hot air washing. For this approach, the protective equipment is placed in a room or chamber and bathed in 120 to 250° F dry air. The washed air is then exhausted through filter banks of activated charcoal. Air washing appears to require approximately 24 to 48 hours to be effective, depending on the type and extent of contamination. 15 20

          The effective cleaning of highly porous materials also continues to be a problem in the industry, as they are not easily decontaminated in a cost effective manner using available techniques. Consequently, contaminated equipment and materials with porous surfaces are many times simply discarded.

25           Other products used to clean and/or decontaminate surfaces include an iodine-containing disinfectant (e.g., I D 0 disinfectant (WINSOL LABORATORIES, Seattle, Washington)). This product is relatively non-adherent, and therefore does not provide a visible means for easily monitoring surfaces that have or have not been cleaned.

30           For use in decontamination procedures, currently available materials and techniques fail to indicate areas that have been treated and/or cleaned. Risk of human exposure to potentially hazardous substances thus exists even after conventional cleaning and decontamination procedures are carried out. Surfactant-water solutions typically used in

cleaning and decontamination applications have poor wetting characteristics, especially when the surface being tested is constructed of polyolefins and/or Teflon® analogs. Additionally, currently used detergent and water preparations frequently contain foam enhances, thus potentially causing problems with disposal and destruction of contaminated suds. Soap foams may also mask areas that have not yet been cleaned.

A cleaning product is needed that will clean easily and rapidly, and that provides an indication of areas that are missed in the cleaning process without the use of ancillary pieces of equipment. In response to growing demands for "green" (i.e., biocompatible) cleaning agents and processes, a need also exists for a cleaning agent that is not in itself a biohazard.

A cleaning composition that is relatively independent of pH changes is also needed to provide enhanced stability of the product, particularly over long-term storage without loss of cleaning efficiency.

It is an object of the invention to provide improved methods for cleaning and/or decontaminating surfaces. More specifically, it is an object of the invention to provide a method of cleaning that employs a cleaning agent having a visually detectable coloring agent and a surfactant, where removal of the visually detectable coloring agent defines areas where the intended scrubbing or cleaning process has been performed. It is also an object of the invention to provide materials and methods useful in teaching decontamination/cleaning procedures. Such may be accomplished with an adherent decontamination/cleaning agent that includes a visually detectable coloring agent.

An additional object of the invention is to provide a technique for detecting punctures or breaches of a protective barrier. Such would be particularly useful in specific exposure -conditions where it is necessary to determine the integrity of a protective garment after exposure or a suspected exposure to a hazardous material. The result of this inspection may dictate secondary decontamination of the wearer, and therefore may serve the additional object of monitoring worker risk of exposure to hazardous materials.

It is a further object of the invention to provide an improved cleaning and/or decontaminating composition having enhanced adherent characteristics. It is still another object of the invention to provide a method to evaluate and compare decontamination

procedure effectiveness with the unaided eye, i.e., a method that may be used under normal white light.

### SUMMARY OF THE INVENTION

5           The present invention satisfies at least one of the above and other useful objectives. Both compositions and methods of employing said compositions in a variety of applications, and particularly, as an aid in methods for cleaning and/or decontaminating a surface, are disclosed. The unique characteristics of the compositions include the presence of a visually detectable coloring agent and its ability to adhere to many different  
10 types of surfaces, including Teflon®.

          The present formulations, in some embodiments, provide an adherent cleaning composition comprising a polymer, an aliphatic alcohol, or a mixture thereof; a visually detectable coloring agent; and a surfactant, the composition being further described as maintaining a constant color upon contact with a contaminant or change in pH. The  
15 cleaning efficiency is also independent of the pH of the composition. The compositions provide for the detection of contaminant without a change in color of the composition and without the necessity of a pH change. The visually detectable coloring agent of the composition is further defined as imparting a color to the composition that does not change upon contact with a contaminant or change in pH.

20           The disclosed compositions, with the visually detectable coloring agents, particularly dyes and pigments, may also include a variety of active ingredients, including but not limited to surfactants, therapeutic agents, biocides, or a combination of all or some of these.

          Where a surfactant is at least one of the active ingredients included, a uniquely  
25 efficacious cleaning formulation is created that gives the user an easily and readily detectable reference in cleaning and/or decontaminating a given surface. A variety of different surfactants may be employed singularly in or combination in the compositions. By way of example and not limitation, such surfactants include ethoxylate alcohol (for example, sulfated ethoxylate alcohol), sulfonates, alkyl sulfates, sulfosuccinates,  
30 alcanolamides, fatty acid esters, ethoxylated triglycerides, cocamido propyl betaines, imidosolines, ethoxylated fatty amines, and the like. In particular embodiments, the surfactant is a sulfated ethoxylate alcohol, such as Witcolate™.

In some embodiments, the composition also includes a polymer, a hydroxylated aliphatic alcohol, or mixtures thereof. By way of example and not limitation, such polymers include polyethylene glycol, polypropylene glycol, glycol ethers, N-methyl pyrrolidone, or any other water soluble polymer that is liquid at room temperature. Examples of hydroxylated aliphatic -alcohols that may be included with the composition include glycerol, ethylene glycol, butane diol, hexane diol, hexane triol, and the like.

In some embodiments, the composition also includes a solvent and an emulsifier. By way of example, the solvent may comprise limonene, and particularly d-limonene. Other solvents, such as aliphatic-aromatic hydrocarbons, alcohols, esters, ketones, aldehydes, amides, glycols, glycol esters, lactones, pyrrolidones, carboxylic acids, as well as halogenated derivatives thereof, may be used in the compositions of the invention alone or in combination.

In some embodiments, the emulsifier may comprise a polymer. The polymer may be either water soluble or water insoluble. By way of example and not limitation, these water soluble polymers include carboxymethyl cellulose, plant gum, polyvinyl pyrrolidone, polyvinyl alcohol, polyethylene oxide, alginates, pectin, gelatin, polyacrylamide, polyacrylic acid, polyethylene glycol, polypropylene glycol, starches, or analogs as well as derivatives thereof. One embodiment of the composition includes the water soluble polymer, sodium carboxymethyl cellulose as the emulsifier.

Other embodiments of the invention may further include an extender. By way of example, such extenders may comprise polyethylene glycol (PEG), polypropylene glycol, glycol ethers, n-methyl pyrrolidone, or mixtures thereof. Other extenders, and more specifically other polymers, may also be used in the composition either alone or in combination with other extenders.

The visually detectable coloring agent of the invention may comprise a synthetic pigment, an organic or inorganic pigment, a plant-based pigment, a dye, as well as mixtures of these agents. In some embodiments of the invention, the visually detectable coloring agent is a colored pigment. Such pigments, by way of example, may be fluorescent (such as T-15 Blaze Orange™. These, and other fluorescent pigments, are available in a variety of colors, including white and orange, and may be used in various embodiments of the composition. Both florescent and nonfluorescent pigments may be

used in the practice of the present invention, and will provide the adherent, readily visible and detectable preparation disclosed.

The compositions of the present invention may further comprise water or some other suitable liquid diluent or carrier. By way of example, this diluent may comprise  
5 from about 0.1%/wt to about 99%/wt of the composition. In some embodiments, water constitutes about 90% to about 95%/wt of the composition.

In one embodiment, the composition comprises from about 1% to about 90%/weight surfactant, about 0.1% to about 90%/weight solvent, about 1% to about 80%/weight emulsifier, about 0.1% to about 90%/weight visually detectable coloring  
10 agent, and about 20% to about 95%/weight polymer. In even further defined embodiments, the composition comprises about 5% to about 25% surfactant, about 2% to about 25% solvent, about 2% to about 25% emulsifier, and about 2% to about 25% visually detectable coloring agent, and about 30% to about 70%/weight polymer.

An even further embodiment of the composition, comprises about 18% to about  
15 22% surfactant, about 12% to about 18% solvent, about 10% to about 15% emulsifier, about 10% to about 15% visually detectable coloring agent, and about 40% to about 50% polymer, such as water soluble polymer.

In another embodiment, the composition comprises about 1.2% to about 1.7% surfactant, about 1.0% to about 1.4% solvent, about 1.0% to about 1.4% emulsifier, about  
20 1.0% to about 1.4%/weight visually detectable coloring agent, about 4.1% to about 5.8%/weight polymer (such as a water soluble polymer), and about 88.3% to about 91.7%/weight water. In still another embodiment, the composition comprises about 42% to about 48%/weight polyethylene glycol; about 28% to about 32%/weight surfactant, such as an ionic surfactant such as sulfated ethoxylate alcohol, or non-ionic surfactant  
25 monylphenol ethoxylate; about 10% to about 15%/weight limonene; and about 10% to about 15%/weight carboxymethyl cellulose.

In some embodiments, the composition comprises about 1% to about 5%/wt surfactant; about 1% to about 2%/wt limonene; about 1% to about 5%/wt pigment (such as a fluorescent pigment); about 2% to about 10%/wt polyethylene glycol; and about 1%  
30 to about 10%/wt carboxymethyl cellulose. In some formulations, the surfactant is nonionic, for example nonylphenol ethoxylate, or an ionic surfactant, for example sulfated ethoxylate alcohol.



The compositions of the invention were found by the inventors to be readily adherent to a variety of different materials, including Teflon®, and to have high viscosity.

It is contemplated that the basic ingredients of the claimed compositions may be formulated together with a herbicide (e.g., Round Up™) as a specific active ingredient, either alone or in combination with other active ingredients defined herein. The compositions may include a biocide, such as an insecticide or insect-repelling preparation. Alternatively, the composition may include a combination of a biocide and a surfactant alone as active ingredients, or a combination of a biocide, an insecticide, and a surfactant.

The present invention also provides for improved methods for cleaning a surface. In some particular embodiments, the method employs the composition defined in Table 1 diluted 1:10 in water, wherein the active ingredient is a surfactant. In broadest application, the method comprises exposing a surface suspected of having a contaminating substance to a composition comprising a polymer, an aliphatic alcohol, or mixture thereof; a visually detectable coloring agent; and a surfactant to provide an adherent coloring indication to the surface; and removing the adherent coloring indicator from the surface. In this method, the color of the adherent coloring indicator remains constant upon contact with any contaminant and does not change color with change in pH. In some embodiments, this method may be further described as including a step of wetting the surface, scrubbing areas of the surface that include the visually detectable coloring agent and rinsing the surface. Water may be used to both wet and rinse the surface being cleaned. The user will readily be able to detect areas that have not been thoroughly cleaned by presence of the coloring agent, and therefore may proceed to repeat the cleaning process where the coloring agent remains. The aforescribed method in particular aspects may also employ any of the specific compositions herein described. In particular embodiments, the method employs a composition that includes a polymer, a visually detectable coloring agent, and a surfactant, combined with an extender, an emulsifier and a solvent.

The compositions are contemplated to be useful in a number of different applications. Because the compositions are adherent to a variety of surfaces, they may be utilized in cleaning a number of different types of potentially contaminated pieces of equipment and clothing items. The compositions adhere well to metal surfaces and to non-porous surfaces. By way of example, surfaces to which the described compositions

adhere include butyl rubber, Bitcon™, PVC, knit or aluminized Nomex™, PBI, Kevlar, nitril rubber, neoprene rubber, Saranex™, Tyvek™, fluoropolymers, and CPE fabric. These materials and others to which the composition adhere are further described as follows:

- 5           •       **Challenge 5000, 5200, 5800, 6400, and X-21** are analog composite materials manufactured by ChemFab, Inc. They comprise an inner and outer layer of a Teflon analog between which is located a layer of fibrous material. The fibrous layer may be woven or non woven. The 5000 and 5200 products are fluoropolymer laminated onto both sides of a woven  
10           Nomex fabric. The 5800/6400 is fluoropolymer laminated on both sides of fiberglass fabric.
- **MIL-C-12189** and **MIL-C-38149C** are materials consisting of butyl rubber.
- **CPE** is a material composed of a woven polyester fabric coated on both  
15           sides with a chlorinated polyethylene formulation.
- **PVC** is a polyvinyl chloride formulation coated onto both sides of woven nylon fabric.
- **Responder** is a plastic made of a polyethylene-based film laminated to both sides of a non-woven polypropylene fabric.
- 20           •       Butyl-coated nylon is an elastomer made of a butyl rubber meeting MIL-C-12189 coated onto both sides of woven nylon fabric.
- Chlorobutyl coated Nomex is an elastomer made of chlorobutyl rubber coated to both sides of woven Nomex fabric.
- Saran laminate is a plastic made of polyethylene/EVOH/polyethylene  
25           laminated on both sides of a nonwoven polypropylene scrim.
- Trelchem VPS is an elastomer/plastic combination of neoprene coated onto both sides of woven polyester with a plastic film on the interior surface of a material.
- X21 is a fluoropolymer Teflon®) laminated to both sides of a **fiberglass**  
30           fabric.

By way of example and not limitation, the methods described are effective for the removal of soil, grease, blood, soot, or mixtures thereof, thoroughly and quickly from a

surface. In particular aspects, the visually detectable agent of choice does not stain the surface being cleaned.

The compositions of the method may further include a biocide, so as to provide a technique for both cleaning and as an aid in assisting in the disinfection of a particular surface. Such would be particularly useful in a hospital setting. In other embodiments, the method may include the use of the aforescribed composition that includes an insecticide. Such insecticide containing cleaning compositions are expected to have particular application in the veterinary field, where a dual purpose of both cleaning and delousing may be accomplished. In still other embodiments of the method, the composition may include a herbicide. Such may be particularly useful in the agricultural industry.

The compositions of the present invention have particular application as decontamination and/or cleaning aids for chemical protection garments (encapsulating vapor protective suits, HAZ-MAT suits, splash suits, boots, turn-out gear, coats, etc.). In these applications of the composition, three objects of the invention are served:

- The composition serves as a readily visible indicator under normal white light of the extent of mechanical brushing of the garment.
- The composition functions as a detergent for the removal of polar and non polar materials from the surface of the garment.
- The composition provides a visual indication of penetrations or physical breaches of the protective surface of the garment. This indication may be manifested as a detectable stain between the inner and outer barrier of the garment, or as a stain on the wearers undergarment.

Although in the embodiments of the invention described, the compositions are applied with a sprayer device which atomizes the composition and deposits it on the surface, other application methods are possible, such as (but not limited to) spreading, brushing and squirting. Likewise, other scrubbing methods can be employed, such as, but not limited to, brushing, sponging, and high pressure liquid stream.

As used in the description of the present invention, the term "contaminant" is defined as any unwanted substance or material, and includes, by way of example, dirt, sand, grease, blood, oils, ink, plant and animal debris, soot and the like. Decontamination

as used in the description of the invention, is defined as the removal or safe neutralization of a contaminant from a surface.

The present invention also provides a cleaning kit. In one particular embodiment, the kit comprises a composition as described herein, including a surfactant and a visually detectable coloring agent, and optionally a brushing or scrubbing implement. In other  
5 embodiments, the kit will further include an insert sheet of instructions outlining the particular methods described herein for application of the composition and removal thereof.

### **BRIEF DESCRIPTION OF THE DRAWINGS**

The following drawing forms part of the present specification and are included to further demonstrate certain aspects of the present invention. The invention may be better understood by reference to one or more of this drawing in combination with the detailed description of specific embodiments presented herein.

15 FIG. A-1 illustrates a particular series of steps for using the compositions in a cleaning and/or decontamination application.

### **DETAILED DESCRIPTION**

The compositions and methods of the present invention provide a highly effective  
20 and unique approach to enhancing cleaning and/or decontamination procedures that rely on visual inspection. The compositions find utility as washing aids in the decontamination or cleaning of personnel, personal protective clothing, and equipment that may have been exposed to contaminating materials. Such contaminating materials include, but are not limited to: dirt, soot chemicals, radioactive materials, radioactive wastes, chemical warfare  
25 agents (such as mustard agents, VX, GA, GB, GD, H and HD agents), biological warfare agents, medical wastes, body fluids and the like.

In particular cleaning formulations, the composition comprises a visual disclosing coloring agent, such as a pigment or dye, in combination with surfactants and agents that give body and thickness to the mixture. When applied to a surface by spraying or other  
30 methods, the composition provides a visible indicator under standard white light that "clings", i.e., adheres, to the surface exposed. The readily visual coloring agent allows the worker to insure via immediate visual inspection portions of the surface that are

covered with the cleaning agent and have not been cleaned. Likewise, disturbances on a treated surface that occur during scrubbing provide visual differentiation between scrubbed and unscrubbed areas. During rinsing, absence of the visible agent indicates that an area on which the cleaning or other active agent has been removed. In this manner, the thoroughness of the decontamination or cleaning procedure may be monitored.

Biocides that can be included in the compositions of the invention may be used to improve the methods by which disinfection procedures are conducted, specifically by indicating where a particular disinfectant has been applied. Such may find particular application, for example, in veterinary medicine, such as in monitoring the application of treatments for lice, mites, fleas, ticks, leaches, parasites, and the like.

Specific applications for the disclosed compositions include:

(a) cleaning and/or disinfecting environmental surfaces, (such as floors, walls, counter tops, and interior patient care areas, both stationary and in vehicles, and other surfaces not designed for intrusive contact with the patient or contact with body fluids);

(b) fire department equipment, such as trucks and other vehicles, hoses, SCBA'S, axes, shovels, respirators, helmets, and hand held fire extinguishers;

(c) emergency vehicles, such as police cars, ambulances, and all types of disaster response equipment, on both interior and exterior surfaces;

(d) all types of military equipment, such as ships, submarines, tanks, aircraft, artillery, troop vehicles, weapons, hand tools, and field hospitals;

(e) food preparation, storage, packaging, and handling equipment, such as blanchers, conveyors, elevators, fillers, graders, slicers, sorters, washers, ovens, vats, mixers, coolers, freezers, refrigerators;

(f) hygiene facilities and equipment, such as locker rooms, lavatories, sinks, showers, urinals, bathtubs and laundry storage bins;

(g) hospital areas and equipment, such as operating areas, emergency rooms, patient rooms, bed frames, refuse containers, rooms;

(h) animal cages and quarters;

(i) car washes, for the thorough cleaning of cars, trucks, motorcycles, and other vehicles;

(j) factories, wherever manufacturing, assembly, or production processes require thorough cleaning of surfaces;

(k) cleaning and disinfecting applications that could occur in morgues, crematories, stockyards, cafeterias, restaurants, laboratories;

(l) de-icing aircraft and other equipment and surfaces; and

(m) as an aid in training personnel in the procedure necessary to carry out any of the above applications. Simulant materials of various types can be applied and removed from a surface in clean-up (DECON) training exercises. This is particularly important in hazardous material cleanup training, where actual hazardous materials may not be used. For example, hazardous material cleanup responders spend much more time in decontamination exercises than in actual hazardous material spill incidents. The compositions of the present invention may be prepared containing an innocuous, non active substance in place of an expensive surfactant or biocide, and used in training. Personnel may be trained how to perform a thorough cleaning job with these less costly preparations of the invention in this manner.

Appropriately formulated, the described compositions may be applied directly to people, animals or plants, to rid the human/animal/plant of surface borne contaminants or disease agents. Also, agents such as medicines, fungicides, or pesticides could be added to the formulation in order to free the human/plant/animal of parasites or to treat skin conditions.

Some embodiments of the invention may include additives that make it very visible in normal white light (i.e., fluorescent, ultraviolet, or bright white). As a general characteristic, embodiments of the composition have a thick, sticky quality that makes it moderately hard to wash off. This is again, important in monitoring cleaning thoroughness. The compositions also have an enhanced wetting ability (low contact angle), a characteristic desirable in cleaning/decontaminating surfaces.

Referring now to the drawings wherein like reference numerals designate like or similar parts throughout the several views, there is illustrated in FIG 1-A, by way of example, a surface 1, covered all or in parts by a contaminant 2 is covered with a coating of the composition by means of a sprayer 4 resulting in a visually detectable layer 5 of the cleaning agent. The colored composition on the surface can be visually inspected to insure that all portions of the surface have been covered (FIG 1-B). The colored composition on the surface is scrubbed with a brush 6, which results in a coating of the colored composition with a disturbed appearance 7 (FIG I-C). The coating of the colored

composition with a disturbed appearance can be visually inspected to insure that all portions of the surface 1 have been completely scrubbed (FIG I-D). A rinsing agent 8 is applied to the surface via a sprayer 9 to remove residual of the colored composition and contaminant 10, resulting in a clean surface 11 (FIG I-E).

5 The following examples are included to demonstrate preferred embodiments of the invention. It should be appreciated by those of skill in the art that the techniques disclosed in the examples which follow represent techniques discovered by the inventors to function well in the practice of the invention, and thus can be considered to constitute preferred modes for its practice. However, those of skill in the art should, in light of the  
10 present disclosure, appreciate that many changes can be made in the specific embodiments which are -disclosed and still obtain a like or similar result without departing from the spirit and scope of the invention.

#### EXAMPLE 1 - COMPOSITION WITH SURFACTANT PREPARATION

15 The present example is provided to outline a particular concentrated form (10X) of the composition. While the present example outlines a 10X preparation, the ingredients may be doubled (to make a 20X concentrate) or cut in half (a 5X concentrate) or any other modification of specific component ingredients to form the composition of the invention. Alternatively, the presently described preparation may be used without  
20 further dilution for cleaning or and/or decontamination applications.

Five chemical components are utilized in one embodiment of the product concentrate. A description of these components, their functional class, their purpose, alternative classes, and possible ranges are provided in the following text.

#### 25 COMPONENTS SURFACTANT / EMULSIFIER

Some embodiments of the composition include at least one surfactant. Witcolate ES-370™ (Witco Chemical) is one surfactant that may be used. Witcolate belongs to a class of anionic surfactants known as sulfated ethoxylate alcohols. The ethoxylate portion  
30 of the molecule is composed of three repeating ethoxy units to which is linked an alkyl chain twelve to fourteen carbons in length. The ethoxylate portion of the molecule is

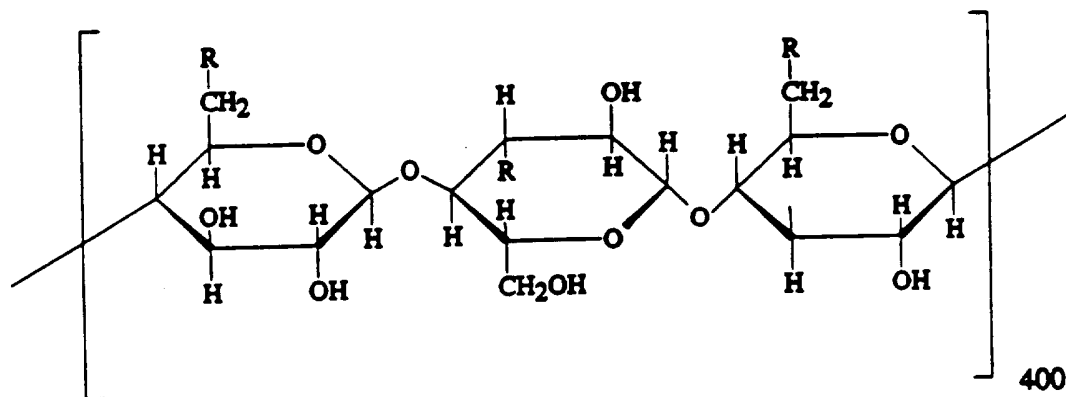




The concentration of the solvent in one embodiment of the composition is about 12.82%/wt. The range for this component in the composition may be about 0.1 to about 90%/wt. A more particularly defined amount of the solvent to include is about 2 to about 25%/wt. Any grade of solvent may be substituted in the preparation of the composition. In addition, it is possible that a wide range of other organic solvents may be substituted for this solvent, including, but not limited to: aliphatic/aromatic hydrocarbons, alcohols, esters, ketones, aldehydes, amides, glycols, glycol ethers, halogenated derivatives of all classes, lactones, pyrrolidinones, carboxylic acids, and the like.

## EMULSIFIER

Some embodiments of the composition include an emulsifier. This component also serves as a viscosity builder. In one embodiment, the viscosity builder/emulsifier is the sodium salt of carboxymethyl cellulose (CMC). CMC is a water soluble polymer. CMC was procured from Hercules/Aqualon as their product 99-7MXF. A grade of CMC that was used in one embodiment of the composition had a molecular weight of about 250,000 and a degree of substitution (carboxylate) of 0.65 - 0.90% (see structure).



$R = -OCH_2COONa$ ,  
with degree of substitution = 0.65-0.90

When included in the composition, the emulsifier component is in suspension in the liquid components. In embodiments of the composition that include water or other suitable diluent, the emulsifier dissolves in the aqueous phase of the mixture. The viscosity of the composition increases to a high degree as a result. The viscosity of the composition allows it to wet low surface energy materials. By way of example, such low surface energy materials include polyethylene and Teflon® derivatives. Because Teflon®

derivatives are used to fabricate chemical protective garments, the compositions are particularly useful in the cleaning and/or decontamination of these materials. In the composition without water or other diluent, viscosity is not as high, making the preparation convenient to measure and dispense. This characteristic of the composition without water or other diluent provides an embodiment that is particularly suitable commercial product, as the composition without water may be stored in relatively small shelf space until time of desired use.

The addition of water will increase further the adherent characteristics of the composition. This adherent character makes the composition particularly suitable for use as a visual means for monitoring the extent of brushing or cleaning of a surface, and as a penetrant of vertical or non-planar surfaces.

Any grade of CMC or other emulsifier may be utilized in the compositions. It is contemplated that emulsifiers having different molecular weights and degrees of substitution from that of CMC will be equally efficacious in the preparation of the present invention.

The amount of the emulsifier, such as CMC, in one embodiment of the composition is about 12.82%/wt. Depending on the grade utilized, the usable range of the emulsifier may be about 1.0% to about 80%/wt.

Other water soluble polymers may be utilized in this formulation as emulsifiers. In particular embodiments of the invention, the polymer should not be soluble in the composition as formulated without water (i.e., the particular solvents included), yet be soluble in water. Possible polymers include, but are not limited to: water soluble modified celluloses, plant gums of all descriptions, polyvinylpyrrolidone, polyvinylalcohol, polyethyleneoxide, alginates, pectins, gelatin, polyacrylamides, polyacrylic acids and its homologs, polyethylene glycols, polypropylene glycols, starches and derivatives.

#### **VISUALLY DETECTABLE COLORING AGENT (PIGMENT/DYE)**

All embodiments of the invention will include a visually detectable coloring agent. In some embodiments, the detectable coloring agent may comprise a colored pigment. Where the composition is to be used as a penetrant to identify breaches or defects in a

surface, the composition would in some embodiments also include a fluorescent material, such as fluorescence.

By way of example and not limitation, a particular pigment used in the compositions is T-15 Blaze Orange™. This pigment was obtained from Day-Glo Color Corp (Austin, Texas). This material is an orange pigment that has fluorescent characteristics in ultraviolet light (down conversion in short, medium, and long wavelengths). The form of the pigment used in some embodiments of the composition is a dry powder.

The pigment or other coloring agent of the composition need not be fluorescent to be useful in the practice of the present invention. As used in the description of the present invention, a pigment is a polymer particle to which a dye has been covalently bonded.

Pigments such as the one described above are available in a variety of colors, all of which can be substituted in this formulation. The pigment or other visually detectable coloring agent functions as a disclosing agent that is visible under normal white light, and aids in identifying the extent of mechanical scrubbing or brushing of a surface, such as that of a garment, or piece of equipment. In addition, pigments may be used that are detectable at very low concentrations in ultraviolet light. In such embodiments, use of the compositions will allow detection of protective barrier penetrations and surface defects that may entrain chemical contaminants.

In some embodiments, the amount of pigment, such as T-15 Blaze Orange™, in the composition is about 12.82%/wt. The range of coloring agent in the composition may be about 1 to about 90%/wt., or at a range of about 2% to about 25%/wt.

Pigment suspensions made in polypropylene glycol (PPG) as well as water dispersed preparations of the pigments were also evaluated. These pigment forms were less suitable in certain embodiments of the composition made without water or diluent. PPG preparations of pigment were found to be soluble in the solvent, d-limonene, thus forming a sticky mass upon the addition of water. Water dispersed pigments, dyes and other coloring agents in water also resulted in less convenient dispensable forms of the composition.

For best results, the inventors used dry powder preparations of the water dispersed pigment such as the dry powder of T-15 Blaze Orange™ pigment. Dry powdered forms of pigments and dyes are available in a wide range of colors, and are readily used in the practice of the invention.

5 Other pigments that may be substituted for T-15 Blaze Orange™ include, but are not limited to: synthetic organic pigments and dyes, plant and animal derived pigments (indigo, porphyrins, etc.) and dyes, inorganic pigments (carbon black, titanium dioxide, metal oxides, metal carbonate, metal sulfate), and dyes.

#### 10 **EXTENDER**

By way of example, particular extenders useful in the invention are polymers and hydroxylated aliphatic alcohols. Polyethylene glycol (PEG) and polypropylene glycol (PPG) are examples of such polymers. PEG has an approximate molecular weight of about 200. This polymer, and other suitable polymers, are available in a variety of  
15 molecular weight ranges from a variety of sources. The PEG or other extender allows the viscosity of the composition to be manipulated to best facilitate ease in dispensing a desired volume, while maintaining suspension of the pigment and viscosity builder.

The concentration of the extender component in one embodiment of the composition is about 46.15%/wt. The amount of this component in the composition may  
20 range from about 20 to about 95%/wt. It is possible for any number of water soluble solvents or water soluble liquid polymers to be utilized in place of the PEG. These may include, but are not limited to: polypropylene glycol, glycol ethers, n-methyl pyrrolidinone, glycerol, ethylene glycol, butane diol, hexane diol, hexane triol, or mixtures thereof. Examples of the hydroxylated aliphatic alcohols include glycerol, ethylene  
25 glycol, butane diol, hexane diol and hexane triol, to name a few.

#### **DILUENT (WATER)**

A diluent, such as water or other solution, may also comprise a component of  
30 some embodiments of the composition. Where included, the diluent may comprise from about 0.1%/wt to about 99%/wt of the composition. In other embodiments, the diluent

comprises from about 50%/wt to about 95%/wt of the composition, while in other embodiments, the diluent comprises between 75%/wt to about 95%/wt of the composition. In one particular embodiment, the composition comprises about 85%/wt to about 95%/wt water or other diluent. The composition including about 92%/wt diluent, particularly water, has been found by the inventors to be particularly preferred and to have especially desirable adherent characteristics to hard-to-wet surfaces, such as Teflon®.

In a particular embodiment, the composition of the invention (without water) was prepared containing the following constituents:

TABLE 1

CLEANING/DECONTAMINATING FORMULATION

<u>Component</u>	<u>Manufacturer</u>	<u>Weight Percent</u>
1. Polyethylene Glycol (200 MW)	BASF	46.15%
2. Ethoxylate Alcohol (sulfated) (Witcolate ES-370™)	Witco Chemicals	15.38%
3. d-Limonene	SCM/Glidco	12.82%
4. T-15 Blaze Orange™	Day-Glo Color Corp.	12.82%
5. Carboxymethyl Cellulose(Sodium) (#7MXF)	Herculese/Aqualon	<u>+12.82%</u>
		<u>99.99%</u>

The described formulation was also prepared with nonyl phenol ethoxylate (a non-ionic surfactant, Tergitol™, Union Carbide) in place of the anionic surfactant, ethoxylate alcohol (sulfated) (See Example 8).

## EXAMPLE 2 - PREPARATION OF READY-TO-USE ADHERENT COMPOSITION

5 The present example is provided to outline the method by which a water  
containing composition of the invention may be formulated from those embodiments of  
the composition ,that do not include water, as well as a method for preparing the  
composition with water or other diluent as an initial composition. Both preparations  
include an amount of water found to provide compositions with particularly desirable  
adherent characteristics and ease of use and application to Teflon®-treated surfaces and  
10 protective garments.

### FROM COMPOSITION FORMULATED WITHOUT DILUENT (WATER):

For use in cleaning applications or decontaminating applications, some  
embodiments of the composition include one part of the composition described in Table  
15 1, or equivalent solvents, pigment/dyes, extenders, surfactants, or emulsifiers, combined  
with nine parts water. This composition is then mixed thoroughly before use. It is also  
expected that mixtures that include .1, .2, .3, .4, .5 to .9 parts, 01 1 to 9 parts water or  
other diluent together with one part of the composition as prepared without water (or  
other diluent) will also provide the described decontamination or cleaning preparation.

### COMPOSITIONS CONTAINING WATER OR OTHER DILUENT:

20 In some embodiments, the composition may also be prepared to include water or  
other suitable diluent as an initial preparation. In such embodiments, polyethylene glycol  
or other extender is not a required component, but may be included as an optional  
25 component.

By way of example, such embodiments would comprise a suspension of the  
following components:

about 1% to about 5%/wt Witcolate ES-370™ (or other surfactant) (particularly  
1.2 to 1.7%/wt);

30 about 1% to about 2%/wt limonene (such as d-limonene) (or other solvent)  
(particularly 1.0 to 1.4%/wt);

about 1% to about 5%/wt T- 15 Blaze Orange™ pigment (or other pigment, dye or visually detectable coloring agent) (particularly 1.0 to 1.4%/wt);

about 1% to about 10% carboxymethyl cellulose (or other polymer) (particularly 1.0 to 1.4%/wt);

5 about 78 to about 96%/wt water (or other carrier solution) (particularly 88.3 to 91.7%/wt);  
and

about 2.0% to about 10%/wt extender (such as PEG or PPG) (particularly 4.1 to 5.8%/wt).

10 Other carrier solutions that may be employed in the practice of the invention include alcohols, ketones, and the like. It is expected that the compositions of the invention in forms with or without the diluent (e.g., water), will be shelf-stable for periods of 1 year or longer.

#### STRIPPABLE FILM COMPOSITION:

15 In other contemplated applications, the carrier solution may comprise a volatile solvent, such as alcohol. In use, the preparation would be allowed to dry on the object treated, and then brushed, scrubbed, or peeled off the surface. The compositions formulated to provide a tough film that is peeled off a surface also include a plasticizer. In any contamination on a surface would be peeled off like a film. No additional liquid  
20 is required to remove the formed film from a surface in this application.

In simplest form, the strippable coating is to be formulated as a pigmented lacquer. This lacquer will include a volatile carrier solvent in which is dissolved a polymer matrix, a plasticization agent, a solvent for the contaminating agent, and a pigment. The solvent carrier may take the form of a mixture of solvents of varying volatility. A graded series  
25 of boiling points are frequently necessary to promote **film** formation. These solvents will have to be selected with respect to the types of materials on which they are going to be applied in order to avoid damaging the surface being cleaned. The solvents should have a low human toxicity and be environmentally acceptable. By example, the solvent system may contain the following solvents: terpenes, alcohols, esters, pyrrolidones, and lactones.  
30 A polymer matrix will be selected which will be soluble in the acceptable solvents. This

polymer should be capable of forming tough, durable films. A variety of polymeric materials may be suitable for this purpose. Some examples of these polymers are: modified cellulosic polymers, acrylic polymers, styrene and copolymers, vinyl polymers and acetates, as well as other classes of elastomeric polymers.

5           It is possible that a polymer blend will be utilized in the formulation to achieve the desired film characteristics. In order to achieve a film which is flexible and removable (non adherent), it will be necessary to utilize plasticizers in the polymer film. Generally, these compounds are oily liquids with a high boiling point, although waxy solids are sometimes employed. These compounds are generally non polar in nature and  
10           may function to solubilize contaminating agents, including those hardened by being dissolved in a polymer. Some examples of these compounds are esters of phthalic acid, esters of benzoic acid, aliphatic hydrocarbons, esters of citric acid. It may be necessary to include additional high boiling components to assure dissolution of the contaminating agent. Finally, the pigment will be incorporated into the composition. The pigment  
15           provides a visual indication of the presence of the film on the surface being cleaned. Additionally, the pigment serves as a film extender, or bulking agent. This will be necessary to facilitate the removal of the film from the surface being cleaned.

          A second approach to the concept of a strippable film is the utilization of a coating which is formulated as a latex, or emulsion. Coatings of this type employ an aqueous  
20           continuous phase with a polymer dissolved in a solvent as the discontinuous phase. Surfactants and stabilizers in the continuous phase promote emulsion formation and stability. The pigment is dispersed in the continuous phase independently of the discontinuous phase. Upon application to the surface to be cleaned, the continuous phase begins to evaporate causing the droplets of the discontinuous phase and pigment to  
25           coalesce, forming a film of relatively high initial viscosity. The solvent in the film is next eliminated through evaporation, forming a continuous solid surface film which can be peeled from the surface. All of the active ingredients utilized in the previously mentioned lacquer formulation would be dispersed in the discontinuous phase with the pigment being dispersed in the continuous phase. The advantages of an emulsion are reduced levels of



volatile solvents, ease of application (lower viscosity formulation), and ease of cleaning application equipment.

### **EXAMPLE 3 - METHOD OF USING VISUALLY DETECTABLE DISCLOSING COMPOSITION**

The present example is provided to demonstrate one particular embodiment of the presently disclosed composition and one preferred method by which it is to be made.

The composition of Table 1 provides an orange-colored concentrate embodiment of the composition which is mixed with water or other diluent in a ratio of 1:10 (concentrate:water) to form a suspension. The resulting suspension was applied to a surface by spraying, such as with a pneumatic or electric sprayer. Alternatively, the composition may be applied by manual means. The surface is next brushed to loosen and suspend any surface contaminants present. After brushing, the surface is to be inspected for evidence of incomplete brushing by visually looking for areas where the colored compositions remain undisturbed. Upon complete brushing and inspection of the surface, the surface is rinsed with a liquid, such as a stream of clean water. The surface may then be inspected for evidence of incomplete removal of the coloring agent. This may be accomplished by visually examining the surface under normal white light for the presence of the visually detectable coloring agent. The process may be determined to be complete when no further visible sign of the coloring agent remains. The device or garment that is so processed may then be easily inspected for signs of any surface area that has not been scrubbed as well as for defects in the surface (e.g., for garments seam failure, barrier perforation, etc.) where signs of the coloring agent may be detected.

### **EXAMPLE 4 - STABILITY STUDY**

The present example demonstrates the shelf stability of the concentrated compositions that do not include water or other diluent.

The composition examined in the present example was prepared according to Table 1. The composition was then stored at room temperature for 90 days. At the end

of that period, water was added to the composition in a ratio of 1:10 (1 part composition, 9 parts water).

The initial (no water) composition was examined for viscosity to determine if it readily poured from its container and was susceptible to accurate measuring. This composition was determined to have a viscosity amenable to easy measurement. The composition was then mixed with water (1:10). This composition was found to adhere well to surfaces of protective garments (Teflon®-like surfaces). The inventors conclude that the compositions are shelf-stable over an extended period of time without any significant loss of adherent capacity when mixed with a diluent.

#### EXAMPLE 5 - WHOLE SUIT TIMED SPRAY TESTS I

The present example demonstrates the utility of the compositions for adhering to a surface and providing a surfactant in an adherent form on a surface. This example also illustrates a visually detectable technique for monitoring areas of contact by visual detection under white light.

The composition used in this example was prepared as defined in Table 1, diluted 1:10 in water, and then applied to a suit of chemical protective clothing using a Wagner® electric sprayer, Model 404. The following summarizes the results of these tests.

The composition was applied to an inflated Lifeguard, Inc. Responder™ Class A fully encapsulating vapor protective suit. Elapsed times were recorded for coating the suit with the composition, for scrubbing the whole suit, and for rinsing all the residue.

Table 2 outlines particular apparatus used in applying the above described composition to a surface here, the surface of a chemical protective suit (Responder® material). The times indicated identify the time at which each step was determined to be completed, visually judged under white light for the presence/absence of the visually detectable coloring agent used.

The test demonstrated that the application method could be carried out in the field in a reasonable period of time, with the composition at a 1:10 dilution performing well as a disclosing agent.

TABLE 2

	Test 1	Test 2	Test 3
Sprayer	Wagner® Electric Model 404	Wagner® Electric Model 404	Goldblatt Pace Setter® Air Sprayer (80 PSI)
5 Spray Time (Full Suite Coverage)	1 min. 52 sec.	1 min. 40 sec.	1 min. 17 sec.
Scrub Time	1 min. 30 sec.	2 min. 0 sec.	2 min. 3 sec.
Rinse Time	1 min. 30 sec.	1 min. 18 sec.	1 min. 29 sec.
Total Elapsed Time	5 min. 12 sec.	4 min. 58 sec.	4 min. 49 sec.
10 Amount of Composition (1:10 dil.) Used	1000 cc	1000 cc	1800 cc
Water Flow In Brush?	No	Yes	Yes
15 Average Height of Water in Tub After Rinse	1"	1/2"	1"
Total Water Used (Scrub and Rinse)	28 gallons	14 gallons	28 gallons

**EXAMPLE 6 - WHOLE SUIT TIMED SPRAY TESTS 11**

The present example was conducted using the composition of Table 1 diluted 1:10 with water.

Table 3 outlines the particular spray devices used, as well as some specific spray, scrub and rinse times found to be useful in actual trials with the compositions to process whole protective suits. The procedure used was essentially as described in Example 5. The results presented in Table 3 demonstrate the utility of the compositions as an aid in cleaning and/or decontamination applications.

TABLE 3

	Trial Test	Test 1	Test 2	Test 3	Test 4
Sprayer	Goldblatt® Pace Setter Air Sprayer	Goldblatt® Pace Setter Air Sprayer	Goldblatt® Pace Setter Air Sprayer	Goldblatt® Pace Setter Air Sprayer	Goldblatt® Pace Setter Air Sprayer
5 Spray Time (Full Suite Coverage)	1 min, 57 sec	1 min, 20 sec	0 min, 53 sec	1 min, 7 sec	1 min, 11 sec
Scrub Time	1 min, 46 sec	1 min, 36 sec	1 min, 16 sec	1 min, 25 sec	1 min, 21 sec
Rinse Time	1 min, 11 sec	0 min, 48 sec	1 min, 2 sec	1 min, 13 sec	0 min, 58 sec
10 Total Elapsed Time	3 min, 54 sec	3 min, 44 sec	3 min, 15 sec	3 min, 45 sec	3 min, 10 sec
15 Amount of Composition (1:10 in water) Used (in cc) (Start/Finish)	1000/1700 (More Composition Added During Test)	2000/1400	2000/1000	2000/1200	1650/950
Water Flow In Brush?	Yes	Yes	Yes	Yes	Yes
20 Average Height of Water in Tub After Rinse	1"	1"	3/4"	1"	1"
Total Water Used (Scrub and Rinse)	28 gal.	28 gal.	21 gal.	28 gal.	28 gal.

## 25 Materials and Methods

### Test 1

30 Responder™ Class A protective suit, Model #50451, Serial #61807. Date manufactured May 25, 1993. Size - Large. Manufacturer - Lifeguard. NFPA 1991. Non-slippery as washed off compositions from treated garment.

Test 2

Same suit as in Test 1, plus Silver Flash Suit worn over the protective suit. Flashmax™ #3, by Chemron, Inc., Order #56958. Aluminized oversuit/flash-fire cover suit.

5

Test 3/Test 4

Lifeguard Responder™ Class B suit, Model #80470, Serial #44651.

## Results:

Foot wet after test 3 - possible leak. Slight orange color on socks. These results demonstrate the utility of the compositions as an aid in cleaning and/or decontamination applications.

10

**EXAMPLE 7 - COMPOSITIONS IN PROTECTIVE CLOTHING PROCESSING**

Some embodiments of the invention provide methods for enhancing washing effectiveness of a surface by providing a visually detectable marker. Such methods find particular application in methods for decontaminating personal protective clothing. Hazardous spill response is a particular unique and useful application of the technology, and serves to provide a more easily detectable system for monitoring the thorough cleaning of protective clothing.

15

For this application, the composition of Table 1 was diluted 1:10 in water. The steps that were followed are defined in Table 4.

20

TABLE 4

	Step
1.	<b>Apply</b> the composition (an electric sprayer is used in this case)
2.	<b>Inspect</b> the surface to assure that it is completely covered
3.	<b>Scrub</b> the surface to clean it. The visual signature of the composition makes it possible to tell scrubbed areas from untouched ones. A brush/handle system that supplies a small, continuous flow of water to the brush is being used to scrub in the photograph.
4.	<b>Inspect</b> the surface to make sure all of the surface has been scrubbed.
5.	<b>Rinse</b> the surface to remove composition and residual contaminants. The brush/handle combination is again used in this example, with the water flow turned to high.

As a last step, the method included inspecting the surface to determine if all traces of the colored composition had been removed.

These steps embody the best mode contemplated by the inventors for processing protective clothing and other surfaces.

#### EXAMPLE 8 - FORMULA WITH NON-IONIC SURFACTANTS

The present example is provided to demonstrate the utility of the compositions claimed with non-ionic surfactants. The particular non-ionic surfactant used in the example is nonyl-phenol ethoxylate.

The specific ranges of PEG, d-limonene, T-15 Blaze Orange™ and CMC defined in Table 1 were used in this formulation. The amount of nonyl-phenol ethoxylate used was 15.38%/wt and the Witcolate ES-370™ (sulfated ethoxylate alcohol) was not included. The formulation was diluted 1:10 in water. Activity of the composition for adhering to a surface of Teflon® poly (tetrafluoroethylene) (PTFE) and a polyethylene laminate, and cleaning ability (waxy contaminant (grease pencil)) were

assessed. The waxy contaminant was easily removable upon application of the composition with minimal scrubbing or rubbing.

#### EXAMPLE 9 - CLEANING EFFICACY

5 On a side by side comparison with common dish washing solution (DAWN®, 3 ounces diluted in 1 gallon water as currently used in the art), the described composition of the present invention (Table 1 composition diluted 1:10 in water) removed a waxy grease contaminant from a Teflon® surface, while the dish-washing solution provided only partial removal after extended scrubbing/rubbing.

10 A combination of non-ionic and anionic surfactants in the composition would also be expected to provide useful cleaning preparations of the invention. In a specific combination, about 7.0%/wt of a non-ionic surfactant, such as the nonyl-phenol ethoxylate of example 8, and about 7.0%/wt Witcolate (anionic surfactant) of may be included in the composition.

15 Mixtures of surfactants, as well as blends of surfactants available to those of skill in the art from commercial sources, may also be used in the practice of the invention. These preparations are also expected to provide effective cleaning and/or decontaminating preparations.

20 The present invention also provides a method for enhancing the adherence and visibility of a cleaning agent on a surface. In one embodiment, this method comprises combining a cleaning agent (i.e., surfactant) with a visually detectable coloring agent (as described herein) and a polymer, a hydroxylated aliphatic alcohol, or a mixture thereof. For example, a cleaning agent could be mixed with a pigment and, as the polymer, polyethylene glycol and/or glycol. These and other  
25 combinations are contemplated in the present invention.

#### EXAMPLE 10 - VISCOSITY

30 The present example provides viscosity measurements of the claimed compositions. Viscosity is expressed in units, as recognized by those of skill in the art. Relative viscosity of a compound provides an objective parameter from which

the adherent character of the preparation may be judged and compared to others. The adherent nature of the claimed compositions is an important characteristic not provided in cleaning and/or decontamination techniques used to date. This characteristic also makes the compositions particularly efficacious in the cleaning of vertical and non-planar surfaces.

5 The present example also provides comparative data on the viscosity of the claimed compositions and the viscosity of compositions used in the art for cleaning and/or decontamination.

The viscosities were determined using a Brookfield spinning disk viscometer. The specific method utilized in the determination of viscosity was drawn from the instructional information  
10 provided by the manufacturer of the viscometer: Brookfield Engineering Laboratory.

TABLE 5

	Dilution	Viscosity (centipoise)
15 Composition	0	196
	1:10 (in water)	1400
Dishwashing detergent	0	296
(Dawn®)	3 oz./1 gal. water	14.8
Winsol®	0	16.4
20	3 oz./1 gal. water	16.3

The viscosity of the diluted form (1:10, water) of the composition of Table 1 was found to be 1400 centipoise. The viscosity of the dishwashing detergent/water at a dilution currently  
25 used for decontaminating a surface was much lower, only 14.8 centipoise.



**Surface Tension Measurements**

Surface tension measurements will be made during optimization of the product. A modified version of American Society for Testing and Materials (ASTM) standardized test D724-89, Standard Test Method for Surface Wettability of Paper (Angle-of-Contact Method) will be used to determine surface tension.

Some embodiments of the compositions of the invention having suitable adherent character are further described as having a viscosity of from about 500 centipoise to about 3500 centipoise. In other embodiments, viscosity range may be about 1000 to about 3000 centipoise. In some embodiments, viscosity is about 1000 to about 2000 centipoise, or even more particularly about 1200 to about 1500 centipoise.

**EXAMPLE 11 - COMBINATION OF COMPOSITIONS  
WITH OTHER PHARMACOLOGICALLY  
ACTIVE AND/OR INDICATOR MOLECULES**

The present example outlines several combinations of the compositions of the invention that may be formulated together with other pharmacologically active components. The following list provides examples of some of these components that may be included in the formulation either individually or collectively for agricultural, veterinary, industrial, and diagnostic applications.

Herbicides (e.g., Round-Up);

Pesticides (e.g., pyrethrins);

Biocides (e.g., iodine/polyvinyl pyrrolidone complex);

Fertilizers (e.g., ammonium nitrate);

Radio isotopes;

medicines (e.g., antibiotics, steroids, aspirin, etc.); and

Fluorescent materials.

These, and many other agents may be combined with the basic composition formulation of the invention in an amount appropriate for the particular application intended by the artisan of ordinary skill.

The following references, to the extent that they provide exemplary procedural or other details supplementary to those set forth herein, are specifically incorporated herein by reference.

5

**EXAMPLE 12**  
**pH Independence of Compositions**

The present example is included to demonstrate that a change in pH of the present compositions does not result in a change in the color of the coloring agent included.

10

The cleaning efficiency of the composition is no dependent on the pH of the compositions, preparations having a highly acidic pH (e.g., pH = 2.79) possessing relatively the same cleaning efficiency as the compositions at a relatively neutral pH (i.e., pH about 6.02) (the pre-mixed preparation). Similarly, preparations having a highly basic pH (e.g., pH = 13.13) possesses relatively the same cleaning efficiency as the compositions while at a relatively neutral pH (e.g., pH about 6.02).

15

1. Pre-mix: Lot number #5178A281

pH = 6.02 (calibrated 400-700)

Color = Bright Orange

20

Composition = 1.77% T-15 Blaze Orange <sup>TM</sup>

0.624% Wilcolate ES-370

1.25% limonene

25

2.70% polyethylene glycol (200 MU)

89.6% water

30

2.29% caboxymethyl cellulose  
(N<sub>a</sub>7MXF)

1.77% Tergitol NP-9

35

2. Pre-mix acidified with citric acid dissolved in H<sub>2</sub>O  
pH = 2.79  
Color = Bright Orange (unchanged) (calibrated 400-700)
3. Pre-mix made basic with KOH (potassium hydroxide)  
pH = 13.12  
Color = Bright Orange (virtually unchanged) (calibrated 700-1000)
4. Basic pH (ph = 13.13) Preparation (as in #3) modified to acidify pH to 8.04 with Pre-Mix (pH 6.02, #4) to achieve a pH of 8.04.  
Color = Bright Orange (unchanged) (calibrated 700-1000)  
pH = 8.04

The present study demonstrated that the color of the cleaning compositions does not change upon manipulating the pH either up (to enhance acidity) or down (to make more basic). The detectable coloring agent thus imparts a color to the composition that remains virtually constant (i.e., retains essentially the same color) with change of pH of the composition, such as between a pH of between about 2 to about 15. The color of the composition is also virtually constant upon contact with a contaminant. The compositions are color-stable, and essentially pH independent. This is in contrast to other cleaning preparations that include coloring agents that are pH sensitive (Casey, U.S. Patent No. 5,110,492), those which are pH-dependent (Jungermann, *et al.*, U.S. Patent No. 3,650,831). The constant color of the present preparations also remains the same upon contact with a contaminant or other material that is of a different pH, such as a surface to be cleaned. This distinguishes the preparations from cleaning preparations that provide for a detectable color change upon contact with a contaminant (Grawe, U.S. Patent No. 5,421,8997).

## REFERENCES

1. "Evaluating the Effectiveness of Haz-Mat Decontamination", David F. Peterson, Fire Engineering, April, 1994.
2. "Personal Protective Equipment Decontamination for Hazardous Waste Operations and Emergency Response", S.Z. Mansdorf, Performance of Protective Clothing: Fourth Volume, ASTM STP 1133, James P. McBriarty and Norman W. Henry, Eds., American Society for Testing and Materials, Philadelphia, 1992.
3. "Standard Operating Safety Guides", Environmental Response Branch, Hazardous Response Support Division, Office of Emergency and Remedial Response, U.S. Environmental Protection Agency, November, 1984.
4. "Haz-Mat Protective Clothing Decontamination Cleaner for Cleaning Has-Mat Suites"™, an advertising brochure distributed by Winsol laboratories, Inc., 1993. (Address: Winsol laboratories, Inc., 1417 NW 51st Street, Seattle, WA, 98107 (800) 782-5501)
5. "IDO Disinfectant"™, Cleaner Disinfectant Sanitizer Winsol Laboratories, Inc. 1993.

WHAT IS CLAIMED IS:

1. An adherent cleaning composition comprising:  
a polymer, an aliphatic alcohol, or a mixture thereof;  
5 a visually detectable coloring agent; and  
a surfactant,  
wherein said visually detectable coloring agent imparts a color to the composition  
that remains virtually constant upon contact with a contaminant or change in pH of  
the composition.  
10
2. The composition of claim 1, wherein the visually detectable coloring agent is  
a colored pigment.
3. The composition of claim 1, wherein the surfactant is a sulfated ethoxylate  
15 alcohol.
4. The composition of claim 1 further comprising a solvent.
5. The composition of claim 4, wherein the solvent is limonene.  
20
6. The composition of claim 1 further comprising a therapeutic agent, a biocide,  
or a mixture thereof.
7. The composition of claim 1, wherein the visually detectable coloring agent is  
25 further defined as a colored pigment.
8. The composition of claim 1 further comprising water.
9. The composition of claim 1 wherein the color of the composition remains  
30 constant at a pH of between about 2 to about 15.

10. The composition of claim 1 having a pH of about 6.

11. A composition comprising:

about 42% to about 48%/wt polyethylene glycol;

about 28% to about 32%/wt surfactant;

about 10% to about 15%/wt colored pigment;

about 10% to about 15%/wt limonene; and

about 10% to about 15%/wt carboxymethyl cellulose,

wherein said colored pigment imparts a color to the composition that remains  
virtually constant upon contact with a contaminant or change in pH.

12. A composition comprising:

about 1% to about 5%/wt surfactant;

about 1% to about 2%/wt limonene;

about 1% to about 5%/wt colored pigment;

about 2% to about 10%/wt polyethylene glycol; and

about 1% to about 10%/wt carboxymethyl cellulose,

wherein said colored pigment imparts a color to the composition that remains  
virtually constant upon contact with a contaminant or change in pH.

13. The composition of claim 11 or 12, wherein the surfactant is a nonionic  
surfactant or an ionic surfactant.

14. The composition of claim 11 or 12, wherein the surfactant is nonylphenol  
ethoxylate.

15. The composition of claim 11 or 12, wherein the surfactant is sulfated  
ethoxylate alcohol.

16. The composition of claim 11 or 12, wherein the fluorescent colored pigment is T-15 Blaze orange.

17. A method for cleaning a surface comprising:

5           applying the composition of claim 1, 11, or 12 to the surface to provide an  
              adherent coloring indicator to the surface; and  
              removing the adherent coloring indicator from the surface,  
wherein the color of the adherent coloring indicator remains virtually constant with  
pH change.

10

18. The method of claim 17, wherein the surface is Teflon®, butyl rubber, Bitcon®, PVC, knit or aluminized Nomex®, PBI, Kevlar, nitril rubber, neoprene, Saranex®, Tyvek®, fluoropolymers, or a CPE fabric.

15           19. A method for enhancing the adherence and visibility of a cleaning agent  
              comprising combining a cleaning agent with the composition of claim 1, 11 or 12.

20. A cleaning kit comprising a composition as defined in claim 1, 11, or 12.

20           21. The cleaning kit of claim 20 further defined as comprising a brush.

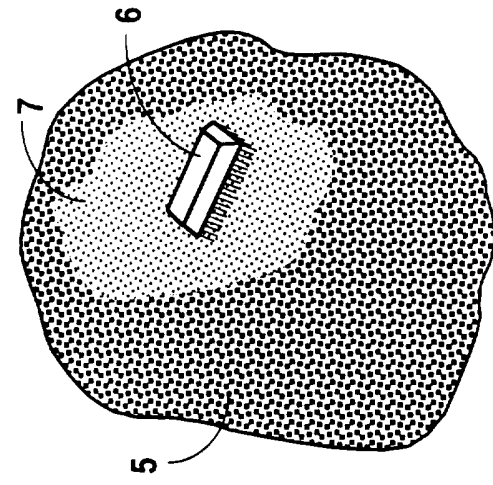


Fig. 1C

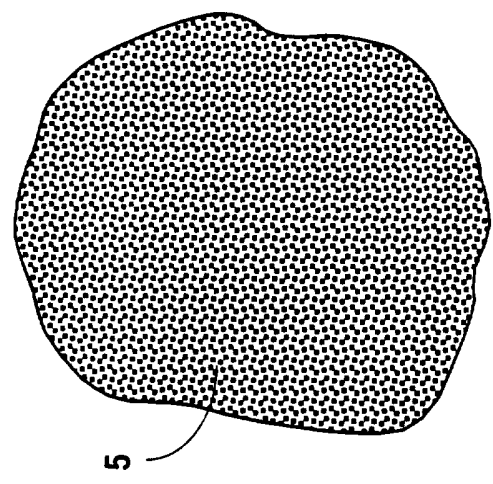


Fig. 1B

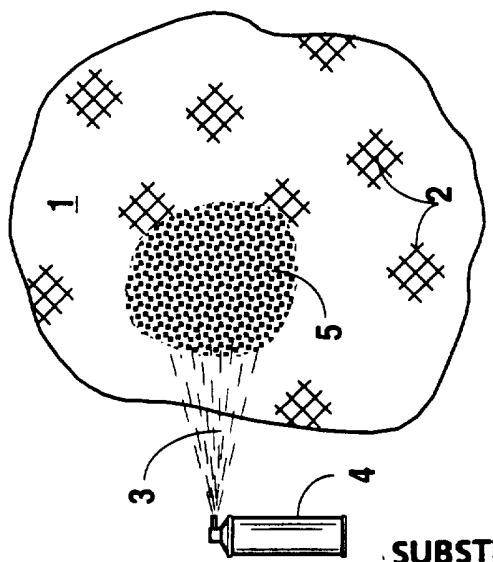


Fig. 1A

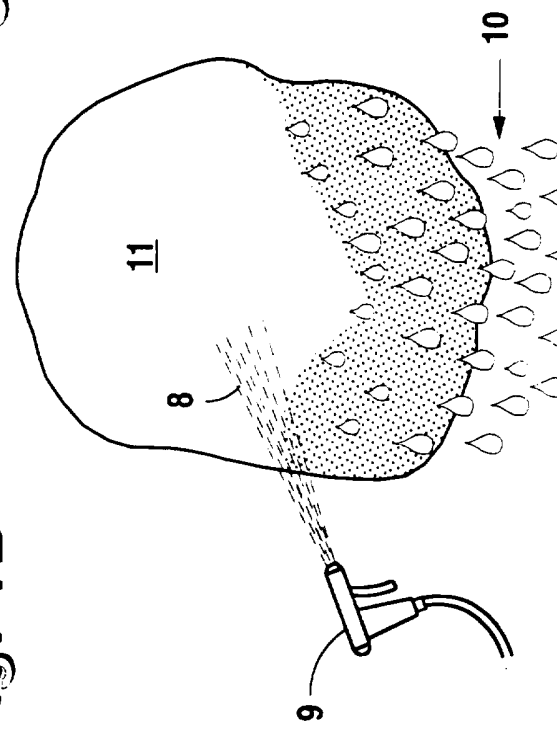


Fig. 1E

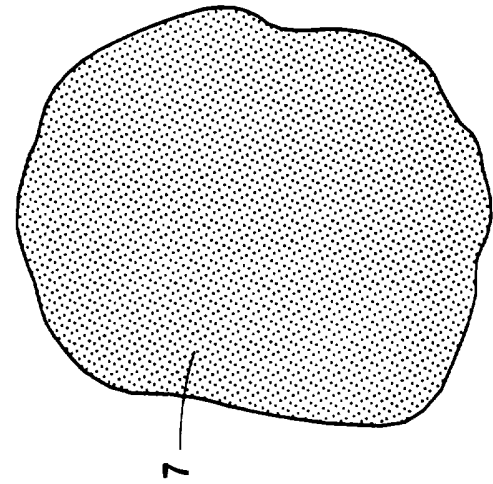


Fig. 1D



**INTERNATIONAL SEARCH REPORT**

International application No.  
PCT/US96/00208

**A. CLASSIFICATION OF SUBJECT MATTER**  
 IPC(6) :C11D 3/14, 3/37, 3/43, 3/44, 3/48, 7/50, 7/60; C23G 1/00, 5/024  
 US CL :Please See Extra Sheet.  
 According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**  
 Minimum documentation searched (classification system followed by classification symbols)  
 U.S. : 134/42; 252/106, 153, 154, 16163, 154, 166, 167, 170, 173, 174.18, 174.21, 174.22, 174.23, 174.24, 174.25, 408.1, 551  
 Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched  
 NONE  
 Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)  
 NONE

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 3,355,392 [CANTOR ET AL] 28 November 1967. See entire document.	1-41
Y	US,A, 3,650,831 [JUNGERMANN ET AL] 21 March 1972. See the abstract, the EXAMPLES and the claims.	1-41
Y	US, A, 3,929,406 [FARMER ET AL] 30 December 1975. See the abstract, the EXAMPLES and the claims.	1-41
Y	US, A, 5,110,492 [CASEY] 05 May 1992. See the abstract, the EXAMPLES and the claims.	1-41
Y	US, A, 5,213,624 [WILLIAMS] 25 May 1993. See the abstract and the claims.	1-41

Further documents are listed in the continuation of Box C.  See patent family annex.

* Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be of particular relevance	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E" earlier document published on or after the international filing date	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"A" document member of the same patent family
"O" document referring to an oral disclosure, use, exhibition or other means	
"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search 09 APRIL 1996	Date of mailing of the international search report <b>26 APR 1996</b>
--	--

Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer <i>M. Barbara Reitz</i> fw DENNIS L. ALBRECHT Telephone No. (703) 308-2525
---	---

**INTERNATIONAL SEARCH REPORT**

International application No.  
PCT/US96/00208

**A. CLASSIFICATION OF SUBJECT MATTER:**

US CL :

134/42; 252/106, 153, 154, 16163, 154, 166, 167, 170, 173, 174.18, 174.21, 174.22, 174.23, 174.24, 174.25, 408.1, 551