VOLATILE CLEANING COMPOSITION

Filed March 23, 1970 2 Sheets-Sheet 1

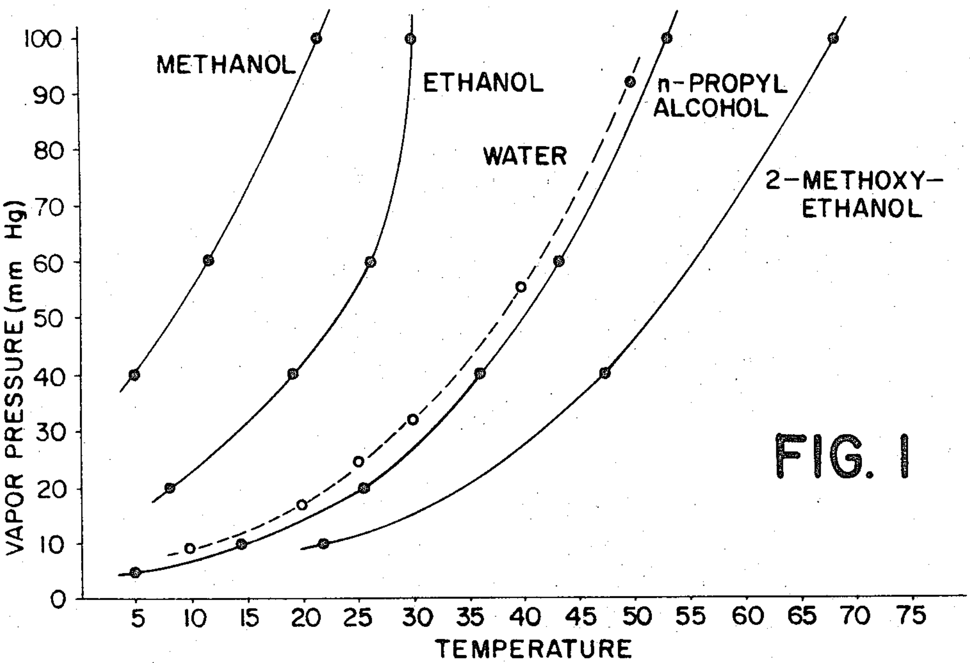


FIG. 1

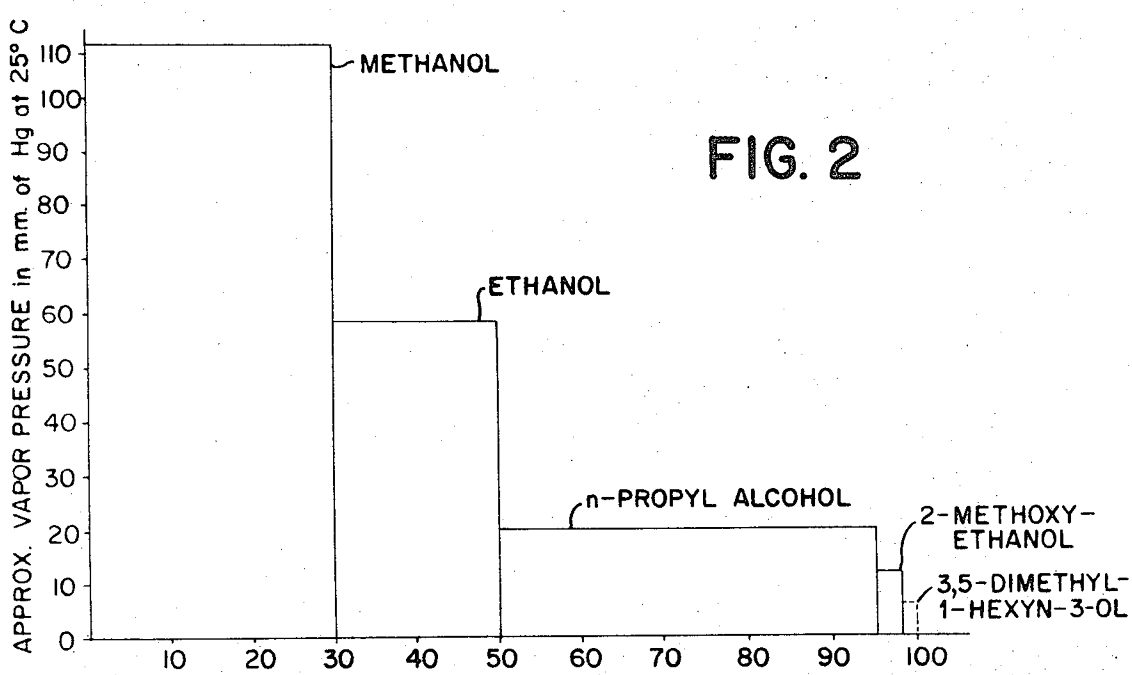


FIG. 2

INVENTORS
GEORGE M. STEPHENSON
JOHN FRANGOS

BY

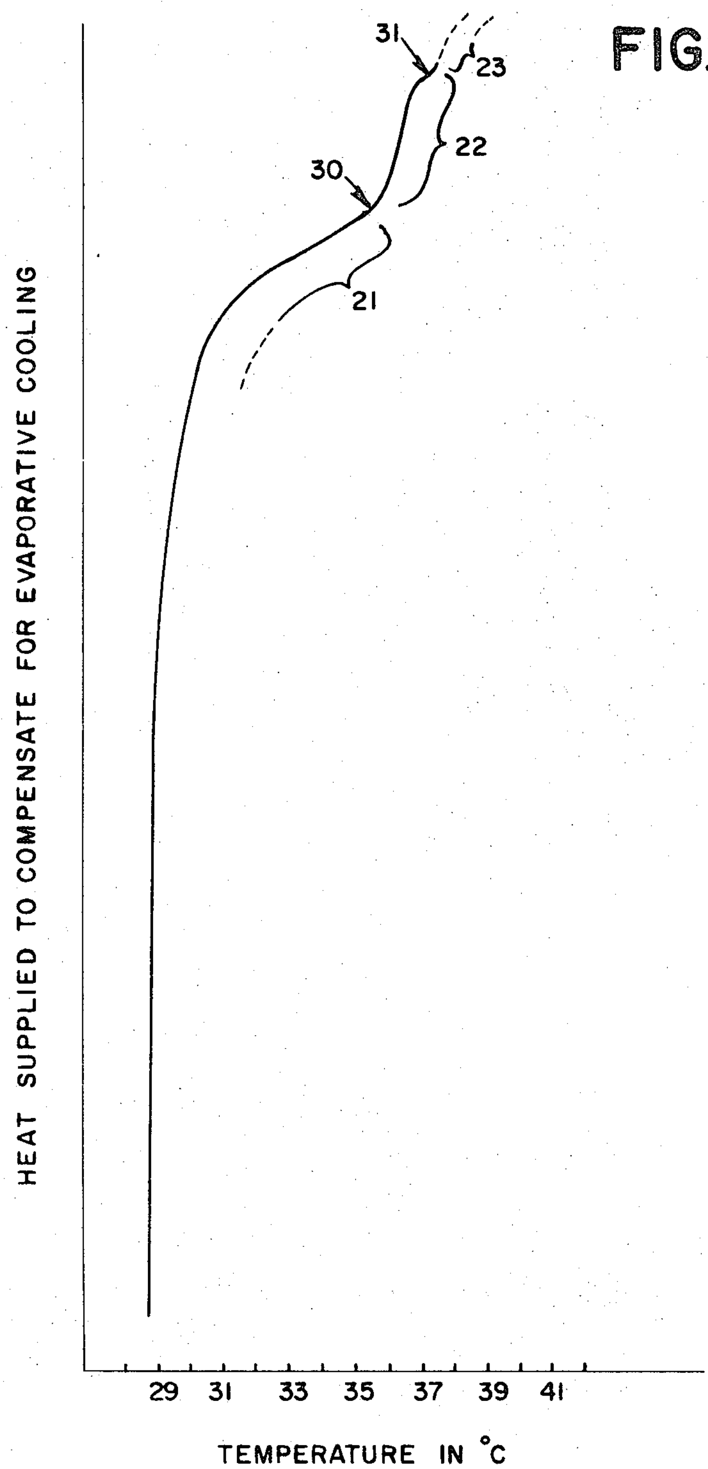
MATTERN WARE & DAVIS
ATTORNEYS

April 17, 1973 G. M. STEPHENSON ET AL 3,728,269

VOLATILE CLEANING COMPOSITION

Filed March 23, 1970 2 Sheets-Sheet 2

FIG. 3



United States Patent Office

3,728,269

Patented Apr. 17, 1973

1

3,728,269

VOLATILE CLEANING COMPOSITION

George M. Stephenson, Wilton, Conn., and John Frangos, New York, N.Y.; said Frangos assignor to said Stephenson

Filed Mar. 23, 1970, Ser. No. 21,725
Int. Cl. C09d *9/04;* C11d *2/50;* C23g *5/02*
U.S. Cl. 252—171 1 Claim

ABSTRACT OF THE DISCLOSURE

A new surface cleaner that is substantially completely volatile, requires no surface rubbing, can be utilized in a pressurized container, a degreasing or ultrasonic cleaning tank, a pump dispenser or other liquid delivery and dispensing systems, and comprises two or more readily volatilizable alcohols in solution with a surface active agent and a grease solvent. When packaged in pressurized containers or pump dispensers, the cleaner is capable of being sprayed as a liquid onto the surface to be cleaned, cleaning that surface, and completely evaporating, thereby eliminating the need for rubbing or wiping the workpiece surface and making the cleaner particularly applicable to optical lens surfaces. The force generated in expelling the formulation from the pressurized container spreads the formulation over the surface, loosening and dislodging particles of dirt and dust thereon while dissolving oils, waxes, salts—organic and inorganic surface contaminants. The volume and velocity of the delivered spray sluices and carries away all dislodged and dissolved materials to the lowest point on the surface being cleaned, leaving only a thin uniform film of the cleaning formulation evenly spread over the surface, which then evaporates substantially completely, leaving no visible residual film or spots. When the cleaning composition is contained in a degreasing or ultrasonic cleaning tank, the surface to be cleaned is dipped into the solution and, upon withdrawal of the surface from the tank, the cleaner will evaporatively run off carrying away the dissolved organic and inorganic substances with it, leaving a clean, residue-free surface.

BACKGROUND OF THE INVENTION

The present invention relates to new, surface cleaning compositions and more particularly to volatile, non-rubbing, optical lens cleaners, packaged in suitable containers.

Many different cleaning formulations have been used in the past for cleaning surfaces such as glass, walls, woodwork, plastics, etc. These compositions generally contain a detergent. After being applied to clean a surface such as glass, generally by scouring, rubbing or abrasive action, they leave a film behind which acts as a trap for dirt and dust particles, and interferes with the optical preciseness of finely ground lenses.

Many attempts have been made to produce volatile cleaning or detergent compositions which will completely evaporate after performing the cleaning function and will leave no residual film. Volatile compositions which leave no residual film are available; however, they are not capable of adequately cleaning substantially all types of contamination that may be found on the surface of a workpiece.

Furtherfmore, most volatile cleaning compositions contain a combination of alcohol and water. Because of the high surface tension of water, these cleaning compositions, after spreading over the surface to be cleaned, coalesce in small droplets or globules. As the solvent evaporates from the surface to be cleaned, the water-soluble

2

dirt concentrates in these droplets and causes surface rings, or spotting.

The limitations of presently existing surface cleaners create severe problems in cleaning finely ground optical lenses, and other surfaces and materials. Because of the high standards to which these lenses are manufactured and the precision required in their use, both rubbing and spotting should be completely avoided. If an optical lens is rubbed even by delicate fibers, fabrics or tissues during the cleaning process, there is a possibility that the surface will be scratched by an entrained abrasive particle and thus the lens efficacy will be reduced. Furthermore, if residual films or spots remain on an optical lens after cleaning, designed light transmission and refraction characteristics are degraded.

Therefore, an object of this invention is to provide a cleaning composition having effective cleaning capability, while leaving no residual film on the surface after cleaning.

A further object of this invention is to provide a cleaning composition which will evaporate substantially completely after its cleaning function has been performed.

Another object of this invention is to provide a cleaning composition that completely eliminates the need for rubbing.

A further object of this invention is to provide a cleaning composition that can be easily applied to any surface by means of various liquid delivery and dispensing systems, such as pump dispensers.

Another object of this invention is to provide a cleaning composition that spreads over the entire surface to be cleaned in a continuous film and thereby eliminates globular or "fish eye" formations and resulting spots.

Another object of this invention is to provide a cleaning composition that can be easily applied to any surface by spraying the composition from a pressurized container.

A further object of this invention is to provide a cleaning composition that contains minimal water.

Another object of this invention is to provide a cleaning composition that is capable of delivery in a high volume stream directed toward the surface to be cleaned, thereby dislodging and carrying away particles of dirt and dust which otherwise could not be removed during the cleaning operation without risk of scratching or marring the lens surface.

Another object of this invention is to provide a cleaning composition that is capable of being applied to many smooth impervious surfaces, such as metals and plastics.

Other and more specific objects will be apparent from the features, elements, combinations and operating procedures disclosed in the following detailed description and shown in the drawings.

SUMMARY OF THE INVENTION

This invention relates to the formulation of a new, completely volatile, low water content cleaning composition comprising varying amounts of two or more readily volatilizable alcohols as the major proportion of its ingredients and at least one volatile surface active agent, and which is utilized in a pressure dispensing or pump dispensing system, in an ambient pressure degreasing or ultrasonic type tank, or in other liquid delivery and dispensing systems. The use of a substantially completely volatile, low water content cleaning composition packaged in a pressurized container provides an extremely effective optical lens cleaner capable of first dislodging and carrying away foreign particles accumulated on the surface and, secondly, capable of dissolving and removing films of the usually encountered types of grease, oil, wax and salt deposits without leaving any residual film or spots. All of the compounds used in the cleaning formulation are substantially volatile and substantially anhydrous.

In a tank, the surface to be cleaned can be dipped or maintained in the cleaning composition to dissolve the organic and inorganic surface contaminants; and, after removal from the tank and evaporation of the cleaning composition, the surface will be adequately clean, residue free and spotless. In this method of use it is desirable to employ two or three such tanks and to transfer the items to be cleaned in the same order from tank to tank so that the final dip will be with a soil-free cleaning composition.

The compositions of this invention achieve these objectives by substantially reducing water and including a volatile surfactant and a volatile grease solvent in addition to different alcohols having different volatility rates to form the major proportion of the cleaning composition, all packaged in a pressurized container having a valve controlled outlet. A cleaning composition is thus created that is capable of being applied at different locations to any surface to be cleaned and which washes away dirt and dust particles upon contact with the surface, dissolves most common inorganic and organic substances, spreads evenly over the entire surface, evaporates in a controlled graduated manner, and thus leaves no visible residual films or spots.

THE DRAWINGS

FIG. 1 is a graph showing the relationship of the vapor pressures of the alcoholic constituents utilized in this invention as a function of temperature as compared to that of water.

FIG. 2 is a diagrammatic representation of the graduated volatility of the components of a preferred cleaning formulation according to the invention.

FIG. 3 is the test results of a Differential Scanning Calorimeter showing the additional heat supplied to the preferred composition of this invention to compensate for evaporative cooling.

SPECIFIC DESCRIPTION

The present invention utilizes a mixture of at least two volatile alcohols and at least one volatile surface active agent. The surface active agent must be miscible or soluble in the alcoholic mixture and may be one or more of the group consisting of wetting agents, detergents or grease solvents. These agents may themselves be readily volatilizable or may be volatilizable as a result of their solution in the alcoholic component.

The alcoholic component employed herein must be readily volatilizable at ambient pressure and temperature, i.e., atmospheric pressure and a temperature of not more than 80° F. Essentially included are methanol or ethanol, or both, which readily vaporize at ambient conditions, methanol being preferred in all compositions as will be pointed out hereinafter. In addition, the propanols (preferably n-propyl alcohol) and unsaturated alcohols of 3–5 carbon atoms may be employed because of the miscibility of such alcohols and the tendency of the mixtures to form highly volatile azeotropes which will evaporate rapidly and completely.

Generally, the alcoholic component will comprise 70–99 percent by weight of the composition and the surface active agents will comprise 1–30 percent by weight of the composition. Of the alcoholic component, at least 30 percent by weight thereof should comprise methanol and/or ethanol; preferably they should comprise at least 40 percent by weight thereof. The preferred alcohols are anhydrous or substantially anhydrous since water is considered detrimental to optimum operation. However, absorption of water in methanol and ethanol is difficult to avoid; accordingly, up to 8 percent by weight thereof and preferably not more than 5 percent may in practice be comprised of water.

If the surface active agents is comprised of components all having relatively low volatility, they should comprise not more than 10 percent by weight of the composition and preferably less than 5 percent. When the surface active agent is comprised at least principally of a component which vaporizes readily at ambient temperature and pressure, it may comprise up to 30 percent by weight of the composition although it is preferably less than 15 percent. The surface active agents include grease solvents such as glycol ethers and dialkyl ethers, both having less than 6 carbon atoms. Detergents having a relatively high degree of volatility which also function as surface active agents to promote uniform wetting are highly advantageously employed; the alkyl-substituted alkyn-ols having up to 12 carbon atoms and preferably 6–9 carbon atoms being exemplary thereof. Other relatively volatile compounds exhibiting surface active propetries may also be employed such as C_1–C_3 esters of C_1–C_4 organic acids.

The preferred embodiment of this cleaning formulation uses three alcohols, methanol, ethanol and n-propyl alcohol, to form 94–98% by weight of the cleaning composition. It is apparent to one skilled in the art, however, that this cleaning composition could be effective by using only two alcohols; preferably, eliminating the ethanol. The inclusion of methanol is important since methanol acts as a solvent for many of the usual inorganic substances on the surface to be cleaned and carries them away by draining. The ethanol is used in the preferred embodiment since it is capable of dissolving both inorganic and organic substances, end serves to provide an evaporative "bridge" between the higher volatility of methanol and the comparatively lower volatility of the n-propyl alcohol. The ethanol will also dissolve inorganic substances and some of the lighter organic substances. The n-propyl alcohol has vapor pressure near that of water and is the last alcohol to evaporate. Since the heavier organic soils are frequently more difficult to dissolve, the use of n-propyl alcohol provides assurance that the stubborn heavier organic substances will be dissolved and carried away by drainage.

The remaining 2–6% of this preferred cleaning formulation comprises 1–3% by weight of a volatile surfactant, capable of performing as both a wetting agent and a detergent, preferably 3,5-dimethyl-1-hexyn-3-ol, and 1–3% by weight of a grease solvent in the glycol ether family, preferably 2-methoxy-ethanol. The use of a grease solvent provides added assurance that all of the heavier organic substances will be removed from the surface and dissolved in the alcohols prior to their evaporation. The surfactant serves a two-fold purpose since it acts as a detergent which gives the formulation an added cleaning ability, and also performs as a wetting agent to assure that the cleaning composition will completely and uniformly spread over the entire surface to which it is applied.

The preferred embodiment of this cleaning composition is as follows:

TABLE I

	Percent by weight	
	Range	Preferred
Methanol	25-35	30.0
Ethanol	15-25	20.0
n-Propyl alcohol	40-50	46.0
2-methoxy-ethanol	1-3	2.0
3,5-dimethyl-1-hexyn-3-ol	1-3	2.0

In FIG. 1, the vapor pressures of the major constituents in the preferred formulation are plotted as a function of temperature. The vapor pressure of water as a function of temperature is represented by the dotted line. By referring to FIG. 1 and Table 1, it can be seem that 96% of the preferred formulation has a vapor pressure which is greater than or very similar to that of water. Since the volatility of a particular substance is directly related to the substance's vapor pressure, it is reasonable to expect barring the existence of azeotropes, that each portion evaporating from the film of mixed solvents will be richer in the more volatile solvent, thus the last portion evaporating will be richest in the least volatile solvent. However, even if azeotropes were to form causing the less volatile material to be in part volatilized with the more highly volatile material; nevertheless, some degree of stepwise volatilization would take place. The ideal, controlled, graduated volatility of the preferred cleaning formulation as it would evaporate from the surface to which it was applied if no azeotropes were formed is diagrammatically represented in FIG. 2. The step that represents 3,5-dimethyl-1-hexyn-3-ol is shown by a dotted line since the vapor pressure was estimated. In reality the evaporation proceeds in a three-stage process as represented in FIG. 3.

The curve shown in FIG. 3 was generated by warming the preferred formulation in a Differential Scanning Calorimeter, Perkin-Elmer Model DSC–1B. The Differential Scanning Calorimeter contains two pans, a sample pan and a control or empty pan. The device records the heat that must be supplied to the sample pan to maintain both pans at the same temperature.

The curve shown in FIG. 3 was generated by placing the preferred formulation in the sample pan and leaving the second pan dry. Both pans were raised to increasing temperatures at the rate of 1.25° C. per minute from about 29° C. to about 39° C. The curve of FIG. 3 records the additional heat reqlired to raise the sample pan temperature through each temperature increment, as compared to the heat needed to raise the dry control pan by the same increment of temperature. The test results shown in FIG. 3 are thus believed to reflect significant fluctuations in the rate of evaporation of the composition with increasing temperature, and strongly suggest, as do visual tests, that the evaporative process occurs in three successive, recognizable steps or stages. Line segment **21** is a smooth curve until the point of inflection **30** is reached. The slope change there, between line segment **21** and the next line segment **22** apparently represents a change in the heat of vaporization taking place in the pan containing the preferred formulation.

Line segment **22** is a steeper, smooth curve until the next point of inflection **31** is reached at which time line segment **23** follows with a significantly different slope than line segments **21** or **22**. The duration of the test was approximately 8 minutes, but when the solution is spread onto a surface, the liquid depth is much less than it was in the test pan, and the formulation normally completely evaporates in 2–3 minutes.

It is believed that during the first stage most of the methanol evaporates, after carrying off the inorganic substances by gravity flow, and also most of the ethanol evaporates after carrying off inorganic and some organic substances by gravity flow. In the second stage, any remaining methanol and ethanol are believed to evaporate and and the n-propyl alcohol also evaporates after removing organic surface contaminants by gravity flow.

The last stage is charcterized by a visible haze, that appears and then slowly disappears; it is believed that this is the evaporation of the remaining 2-methoxy-ethanol and 3,5-diamethyl-1-hexyn-3-ol. At the end of two to three minutes, the surface is clean, has no dirt spots, and shows no visible residual film.

Even if the proposed three-stage evaporation process does not in fact occur, the judicious selection of solvents and surfactants, which have the capability of dislodging, deterging or dissolving the soils, are substantially volatile and free of non-volatile impurities and therefore do not leave deposits, are substantially water-free and have low surface tensions and different vapor pressures, does create a cleaning composition that is capable of being applied to substantally any surface, either by utilizing a pressurized container or by immersion in an ultrasonic or conventional cleaning or degreasing tank, completely cleaning that surface by removing not only dust and dirt particles but also commonly encountered inorganic and organic contaminants and leaving a residue-free, spotless surface.

Furthermore, the proper selection of one or more volatile solvents whose vapor pressure is less than that of water avoids severe evaporative cooling effects. Generally, upon application of the cleaning composition, the surface of the workpiece is cooled to a temperature below the dew point and causes moisture condensation. By selecting solvents that have vapor pressures less than water and, also, are solvents for water, during the final stages of drying the condensed water will be dissolved in the solvent that are capable of uniformly wetting the surface. This prevents the condensation water from coating the surfaces, during drying, in the form of discrete droplets, or "fish-eyes," with minute traces of particulate matter suspended and dissolved within, and leaving said particulate matter behind after drying in characteristic, spot-lke, visible patterns.

Abrupt evaporative cooling, condensed water droplets, isolated separate "fish-eye" drops of independently evaporating solvent, and deposits or residual films of contaminants and impurities left behind by evaporating solvent are all substantially eliminated by the formulations of this invention, whether or not a step-like evaporation process occurs.

For these reasons, tests have shown the formulation discussed above to be the preferred one for a non-rubbing lens cleaning composition. It is possible, however, to vary the substances used in this formulation and still obtain a cleaning composition that will perform adequately. The ranges over which the preferred substances can be varied are shown below:

TABLE II

	Percent by weight
Methanol	15–50
Ethanol	0–25
2-methoxy-ethanol	0–4
3,5-dimethyl-1-hexyn-3-ol	0–4
n-Propyl alcohol: Comprising the remainder, preferably at least 25%.	

In using this table of ranges it is important to remember the purpose for which each of the substances is included. It would be unwise to formulate a cleaning composition that used the maximum for all the substances and thereby left n-propyl alcohol at its absolute minimum of 17% by weight. The n-propyl alcohol is required to dissolve and remove the heavy organic substances on the surface. Since the heavy organic substances resist dissolution to a greater extent than the inorganic substances, it is imperative that a sufficient amount of n-propyl alcohol is present in the formulation so that removal of all organic substances will be assured. Consequently, when the methanol content is increased to its upper limit, the ethanol content should be decreased.

It would be possible to formulate a cleaning composition that employs the teachings of this disclosure without using all of the compounds suggested herein or by using substites. One example of possible substitutes would be to use other glycol ethers in place of 2-methoxy-ethanol. In place of 3,5-dimethyl-1-hexyn-3-ol, any other volatile surfactant or combination of volatile surfactants which is capable of performing as both a detergent and a wetting agent could be employed. Furthermore, isopropyl alcohol may be substituted for n-propyl alcohol. Small amounts of non-volatile surfactants may be employed provided that they leave continuous optically acceptable film residues, for example dioctyl ester of sodium sulfosuccinic acid. Examples of substitute cleaning compositions are shown in Tables III and IV.

TABLE III

	Percent by weight
Methanol	2.0
Ethanol	16.67
n-Propyl alcohol	79.3
2-butoxy-ethanol	1.0
3,5-dimethyl-1-hexyn-3-ol	1.0
Diocetyl ester of sodium sulfosuccinic acid	0.03

TABLE IV

	Percent by weight
Methanol	2.0
Ethanol	16.67
n-Propyl alcohol	46.0
Isopropyl alcohol	33.3
2-butoxy-ethanol	1.0
3,5-dimethyl-1-hexyn-3-ol	1.0
Dioctyl ester of sodium sulfosuccinic acid	0.03

These cleaning compositions would be suitable substitutes for the preferred formulation in applications where an optically clear film would be permissible.

Extensive testing has been performed using isopropyl alcohol, and it was discovered that isopropyl alcohol left a slight residue upon evaporation. For that reason, isopropyl alcohol has not been included in the preferred embodiment of this cleaning composition. If, however, a residue-free isopropyl alcohol were available, it could easily be substituted for the n-propyl alcohol.

The importance of the advantage that is achieved by providing a formulation for a cleaning composition is substantially free of water was clearly observed in the testing of this formulation, wherein an experimental batch was made which contained 1% by weight of water. After application to the surface to be cleaned and evaporation, it was observed that the surface contained dirt spots. By using the preferred embodiment of this cleaning composition, which is substantially without water, a clean, residue-free, spotless surface was obtained.

The efficacy of this cleaning composition is enhanced by delivery under pressure to provide a soil displacement force by packaging the formulation in a pressurized container or pump dispenser having a valve actuated discharge outlet. In a convenient embodiment the formulation is packaged under pressure in an "aerosol-type" pressurized container with a propellant, preferably dichlorodifluoromethane. This unique system provides a two stage cleaning operation for any surface to which it is applied. In the first stage the particles of dust and dirt are dislodged and sluiced away by the application of the formulation to the surface. This self-flushing action is accomplished by the forces produced in propelling the formulation toward the surface to be cleaned. As the cleaning composition impacts upon the surface to which it is applied, the spray dislodges and sluices away the nonsoluble particles of dust and dirt.

The second cleaning stage is achieved by the cleaning composition itself as it uniformly spreads over the entire surface to which it is applied and dissolves and carries away by gravity flow inorganic and organic substances on the surface, without leaving any visible residual film or spots. This arrangement provides a self-flushing, self-cleaning, completely evaporative, non-wiping lens cleaner, leaving a residue-free, spotless surface.

The cleaning operation is accomplished by remote application and without ever having to touch the surface to be cleaned with anything except the cleaning composition. The tests that have been performed on the surface cleaner formulated according to this invention have proven that normally soiled surfaces can be cleaned by a single application of this surface cleaner without manual contact with the surface. Furthermore, heavily soiled surfaces have also been cleaned by this surface cleaner by only several applications of the surface cleaner, and without manual contact.

These cleaning compositions have been found to be

compatible with many materials. While the cleaning of optical lens surfaces was the principal objective in the formulation of these cleaning compositions, their efficacy is not limited to lens surfaces; the compositions of this invention are highly effective in cleaning many different materials.

The preferred embodiment for the surface cleaner comprises packaging the formulation previously described with dichlorodifluoromethane as the propellant in such a manner that the pressurized container will contain about 85% by weight of the formulation and about 15% by weight of the propellant. This has been prove to be an effective ratio to provide sufficient force for stage one "dislodging" cleaning. The kind and amount of propellant used, however, can be greatly varied since it is dependent upon other surrounding conditions. The propellant content and type of propellant can be varied in accordance with the delivery force desired or the type of spraying effect that is sought. Furthermore, for every different discharge outlet used the propellant may be altered in order to achieve the propelling and spraying conditions desired.

It is important to note that difficulties that have existed in pre-existing cleaning compositions are eliminated by this surface cleaner. This surface cleaner not only dislodges and sluices away surface dirt and dust particles upon application, but then uniformly spreads over the surface to which it is applied, and dissolves inorganic and organic substances. It is believed that as the formulation evaporates, it retreats as a single body of liquid to the lowest point of the surface to which it was applied with the retreating rear edge of this liquid body "pushing" ahead of itself and carrying with it the dissolved contaminants, leaving a clean, residue-free, spotless surface without any requirement for rubbing, polishing or additional or supplemental contact with the surface.

The major difficulties with conventional surface cleaners are the need for rubbing the surface to achieve adequate cleanliness, and the existence of a residual film and spots on the surface after the cleaner has evaporated. Many of these prior art difficulties are eliminated in this invention by using methanol in addition to one or more other readily volatile solvents and substantially completely eliminating the use of water, other than that present as a trace impurity.

Spotting results from the cleaners that contain appreciable water because the high surface tension of water causes the cleaning composition to distribute itself over the surface in numerous globular or "fish-eye," formations during drying. As the cleaning composition evaporates, trace insolubles and particles picked up from the atmosphere become increasingly concentrated in each globule or "fish-eye," and after final drying remain on the surface as dirt spots. By using solvents of low surface tension, the formulation of this invention does not form globules upon application to a surface or during drying, but instead spreads evenly over the entire surface to be cleaned and remains as a substantially uniform film throughout the drying period. Although not completely necessary, the preferred embodiment of this invention includes a wetting agent to further enhance the complete spreadability of this formulation. By eliminating globular or "fish-eye" formation, dirt spots are substantially eliminated.

The second advantage achieved by this invention is the elimination of the need for any rubbing of the surface at all. Rubbing with tissues, swabs, lens cleaning cloths or any other medium is dangerous since there is always the possibility of having an abrasive particle entrained and, thereby, scratching the surface. This is especially hazardous when dealing with optically precise lenses. By using only compounds that are volatilizable at ambient or ordinary room temperatures, the need for surface rubbing is eliminated.

Although some prior art cleaning compositions are theoretically volatile, in actual practice they leave a residual film believed to comprise dissolved and unevaporated materials or impurities on the surface, and this film is capable of trapping dirt and dust particles. The preferred formulations of this invention however are not only volatile, but also avoid leaving residual films after evaporation.

In the formulations of this invention, the alcohols used have graduated proportional volatility rates. After the cleaning composition has completely spread over the lens surface, it appears that evaporation occurs in a controlled, graduated manner. This apparent controlled rate of evaporation coupled with the use of some solvents whose vapor pressure is less than that of water avoids extensive atmospheric moisture condensation as discrete droplets, due to evaporative surface cooling. However, these volatile solvents dissolve the condensed water and maintain it as a uniform surface film preventing spot formations due to soil concentration in the condensed droplets of water. Removal of organic and inorganic substances is further assured by including in the formulation small amounts of a surface active agent and a grease solvent.

Since the foregoing description and drawings are merely illustrative, the scope of the invention has been broadly stated herein and it should be liberally interpreted to secure the benefit of all equivalents to which the invention is fairly entitled.

We claim:

1. A surface cleaning composition consisting essentially of 15–50 percent by weight of methanol, 0–25 percent by weight of ethanol, 25–85 percent by weight of n-propyl alcohol, 1–3 percent by weight of 2-methoxy-ethanol, and 1–3 percent by weight of 3,5-dimethyl-1-hexyn-3-ol.

References Cited

UNITED STATES PATENTS

3,245,912	4/1966	White	252—70
3,463,735	8/1969	Stonebraker et al.	252—171
2,386,106	10/1945	Gangloff	252—DIG. 10
2,993,866	7/1961	Vaughn et al.	252—171

FOREIGN PATENTS

714,019	7/1965	Canada	252—DIG. 10
714,521	7/1965	Canada	252—DIG. 10

LEON D. ROSDOL, Primary Examiner

W. E. SCHULZ, Assistant Examiner

U.S. Cl. X.R.

106—311; 252—364, DIG. 10

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,728,269 Dated April 17, 1973

Inventor(s) George M. Stephenson

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, line 40 - delete "evaporatively"
Column 3, line 75 - change "is" to -- are --
Column 4, line 29 - change "end" to -- and --
Column 5, line 55 - delete "and"
Column 6, line 12 - change "solvent" to -- solvents --
Column 6, line 14 - change "condensation" to -- condensed --
Column 7, line 30 - insert --that-- after "composition"
Column 8, line 12 - change "prove" to -- proven --

Signed and sealed this 18th day of December 1973.

(SEAL)
Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

RENE D. TEGTMEYER
Acting Commissioner of Patents

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,728,269 Dated April 17, 1973

Inventor(s) George M. Stephenson

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, line 40 - delete "evaporatively"
Column 3, line 75 - change "is" to -- are --
Column 4, line 29 - change "end" to -- and --
Column 5, line 55 - delete "and"
Column 6, line 12 - change "solvent" to -- solvents --
Column 6, line 14 - change "condensation" to -- condensed --
Column 7, line 30 - insert --that-- after "composition"
Column 8, line 12 - change "prove" to -- proven --

Signed and sealed this 18th day of December 1973.

(SEAL)
Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

RENE D. TEGTMEYER
Acting Commissioner of Patents